

## **Round-robin validation exercise for the determination of through-thickness shear strength in compression**

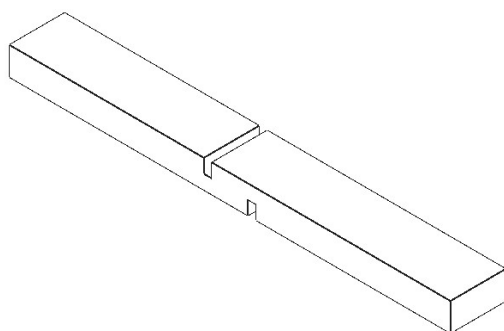
### **Summary**

This Measurement Note details a round-robin exercise undertaken in order to provide precision data to support the proposal of the double notch shear test in compression as a new work item for ISO test methods.

The exercise involved the manufacture of specimens by six different participants, using three different materials. Subsequent testing of all specimens was performed at NPL. Statistical analysis was performed on the test results according to ISO 5725-2, using 95% confidence limits, to determine the method's repeatability and reproducibility.

The repeatability and reproducibility values were generally high due the sensitivity of the test to specimen preparation.

The work was carried out as part of the "Measurements for Materials Systems" (MMS) programme funded by the Department of Trade and Industry.



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## Introduction

An inter-laboratory validation exercise has been undertaken using the double notch shear test in compression, for the determination of the through-thickness shear strength of polymer matrix composites. The purpose of this work was to obtain precision data to support the progress of this test method as a proposal for an ISO new work item (NWI).

The exercise was conducted using six participating organisations and three materials. Each participant manufactured the specimens and sent them back to NPL for testing. The results were then analysed according to ISO 5725-2<sup>[1]</sup> using 95% confidence limits.

The materials investigated were:

- Material 1 – Unidirectional carbon fibre/epoxy, autoclave moulded (CFRP)
- Material 2 – Glass fibre/polyester, mixed format pultrusion (PUL)
- Material 3 – Woven glass fibre/epoxy, press moulded

The proposed method is similar to that in ASTM D 3846-94<sup>[2]</sup> but has been modified to reduce the specimen width from 12.7 mm to 10 mm. This ensures equivalence with both the short beam shear method<sup>[3,4]</sup> and other compressive test methods<sup>[5]</sup>. The new specimen dimensions are shown in Figure 1.

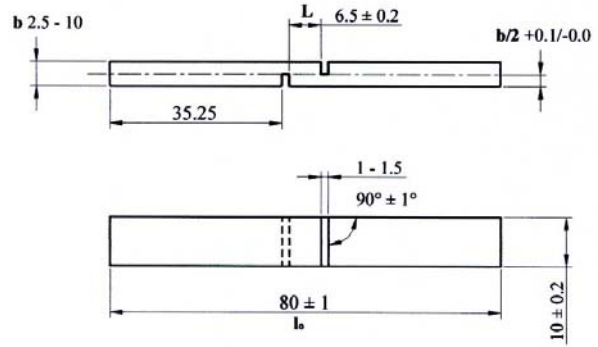


Figure 1 - Double notch shear specimen dimensions.

The double notch shear test consists of a notched specimen held in an anti-buckling fixture, which is based on the ASTM D 695<sup>[6]</sup> fixture. This modified version is shown in Figure 2. The specimen is supported along its entire length, with the assembling screws secured finger-tight, 0.1 Nm, to allow free movement along the vertical axis. The specimen is then end-loaded in compression at a displacement rate of 1 mm/min whilst held in the fixture.

The through thickness shear strength,  $\tau$  (MPa), is then calculated using the equation:

$$\tau = \frac{F_{\max}}{b \times L}$$

where:

- $F_{\max}$  is the maximum load, (N);
- $b$  is the specimen width, (mm);
- $L$  is the axial distance between notches, (mm).

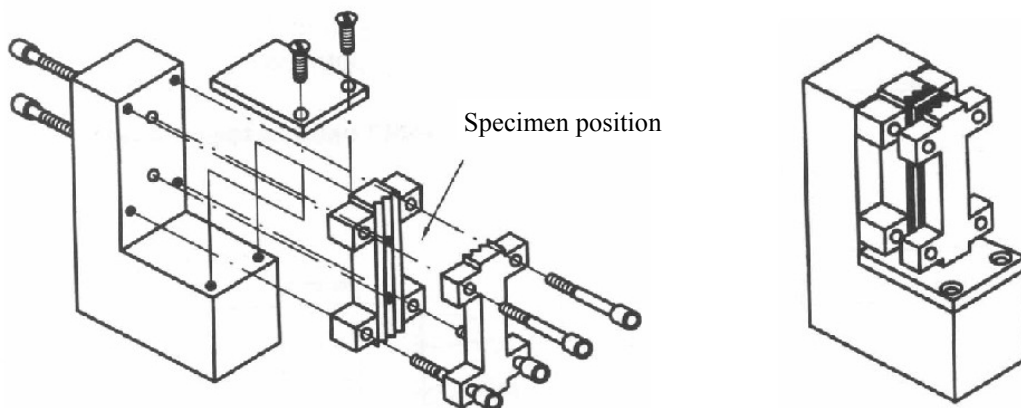


Figure 2 - Schematic of anti-buckling fixture.

Failure of the specimen occurs in shear between the two parallel notches. These are machined to a depth equal to half the specimen thickness and spaced equidistant from the specimen mid-length on opposing faces. The cross-head displacement and load are monitored and recorded throughout the test.

Previous round-robins led by NPL have highlighted the importance of specimen manufacturing quality as the primary factor contributing to inter-laboratory differences for this type of test, rather than the usual test operator dependence. This round-robin concentrated on the specimen machining aspects of the method, with all testing being conducted by a single operator at NPL.

### Round-robin procedure

Coupon panels of the three materials were sent to participants to be machined according to a draft test procedure, written by NPL. Participants were requested to machine a minimum of five specimens per material and fill in a report sheet detailing the machining techniques used.

The specimens were then returned to NPL for detailed measurement and testing by a single operator following the same draft test procedure.

The decision to conduct the round-robin in this manner was taken for two reasons:

- The actual test was considered to be straight forward. The critical aspect was considered to be the machining precision of the specimen notches in order to achieve the required failure mode. Also previous round-robins<sup>[7]</sup> showed that the main cause of variability was differing specimen manufacture processes and that the impact of different operators was less important.
- It was thought that the ASTM D 695 anti-buckling fixture would not be widely available to many of the participants and that sending a single fixture to each in turn would prove to be too time consuming.

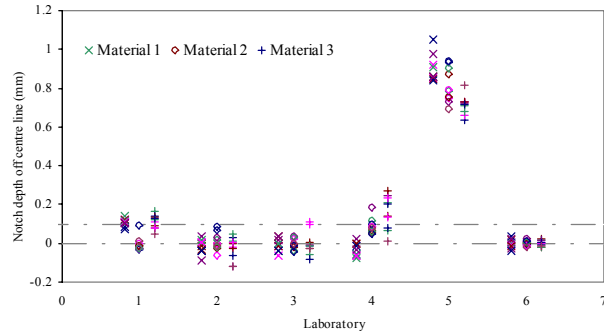


**Figure 3 - Notch depth relative to specimen centre line: grey line represents tolerance limits.**

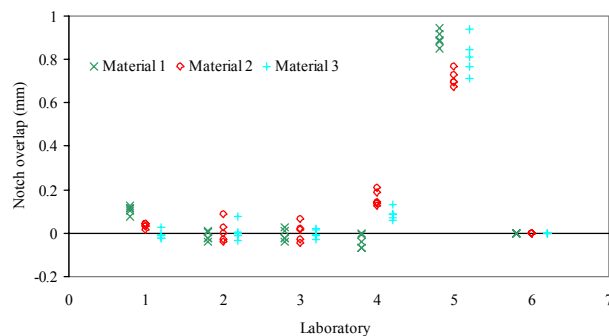


**Figure 4 - Measurements of notch overlap.**

### Manufacture and testing

Details of the manufacturing process adopted by different laboratories are given in Table 1. This information was collated from report sheets returned by participants.

**Table 1 - Variations in specimen machining.**

| Machining process / Cutting tools                                        | Speed (rpm) | Feed Rate (mm/min)           |
|--------------------------------------------------------------------------|-------------|------------------------------|
| Vertical milling machine<br>High speed steel end mill $\varnothing$ 1 mm | 800         | Manual feed<br>$\approx$ 120 |
| Circular / plate saw<br>PCD 40 grit wheel                                | 1800        | 800                          |
| Surface grinder<br>Standard pink wheel grade PA 60                       | 2700        | 7500                         |

Critical dimensions of the specimens were measured at NPL prior to testing. The spacing between the notches, the notch widths and notch depths were measured using a travelling microscope, and other dimensions were measured with a micrometer having a resolution of 0.005 mm. Measurements of the individual notch depths are shown in Figure 3, with the zero line denoting the mid-plane of the specimen thickness. Notch pairs from each specimen, are shown in the same colour, and the grey

lines indicating the tolerance levels specified in the procedure. Values above zero represent a notch machined deeper than the centre plane. From this, the notch overlap was calculated for each specimen and these are shown in Figure 4. Typical notch width measurements are shown in Table 2.

**Table 2 - Typical notch widths (mm).**

| Laboratory | 1     | 2     | 3     | 4     | 5     | 6     |
|------------|-------|-------|-------|-------|-------|-------|
| Material 1 | 1.14  | 1.45  | 1.391 | 1.036 | 1.887 | 1.429 |
| Material 2 | 1.153 | 1.513 | 1.461 | 1.116 | 1.947 | 1.520 |
| Material 3 | 1.146 | 1.546 | 1.499 | 1.12  | 1.856 | 1.514 |

A brief investigation into the measured data shows:

- Laboratory 5: notch depths were machined far too deep on all materials,  $\approx 0.85$  mm past the specimen centre plane, with a radiused rather than the square notch specified by the draft procedure.
- All laboratories, with the exception of Laboratory 5: machined the notch width and separation to the required tolerances.
- Laboratory 5: material 1 was not machined to length (10 mm too long).
- Laboratories 3 & 4: not all specimen ends were machined square and additional side

**Table 3 - Original data: Shear strength (MPa).**

| Material<br>Laboratory | 1    |      | 2    |      | 3    |      |
|------------------------|------|------|------|------|------|------|
| 1                      | 67.1 | 62.5 | 36.5 | 36.3 | 58.4 | 60.3 |
|                        | 62.5 | 57.3 | 38.0 | 31.6 | 54.7 | 60.1 |
|                        | 65.0 |      | 38.1 |      | 60.8 |      |
| 2                      | 66.9 | 76.9 | 28.3 | 30.1 | 58.6 | 58.9 |
|                        | 81.0 | 66.4 | 32.3 | 37.8 | 58.7 | 66.1 |
|                        | 70.0 |      | 30.9 |      | 60.2 |      |
| 3                      | 49.6 | 67.7 | 32.6 | 35.8 | 58.9 | 54.7 |
|                        | 79.9 | 62.6 | 30.2 | 35.9 | 56.0 | 49.1 |
|                        | 58.7 |      | 32.5 |      | 55.6 |      |
| 4                      | 75.7 | 46.8 | 36.4 | 34.4 | 56.6 | 57.8 |
|                        | 74.8 | 82.4 | 22.5 | 33.3 | 57.8 | 53.0 |
|                        | -    |      | 29.7 |      | 58.5 |      |
| 5                      | 57.3 | 52.8 | 22.4 | 21.8 | 45.5 | 45.8 |
|                        | 54.3 | 57.9 | 27.2 | 27.4 | 47.7 | 44.0 |
|                        | 54.0 |      | 20.6 |      | 47.0 |      |
| 6                      | 69.6 | 74.3 | 33.2 | 35.9 | 59.1 | 58.1 |
|                        | 82.6 | 68.3 | 36.6 | 34.7 | 62.0 | 59.4 |
|                        | 57.5 |      | 36.3 |      | 59.3 |      |

edge damage was visible on certain specimens.

- Most laboratories, with the exception of laboratory 6: showed difficulties in machining the notch depth to the tolerances set in the draft procedure.

In accordance with the draft test procedure, tests were mechanically conducted using a calibrated Instron 4500 series screw driven test machine. Compression loading of the specimen held in the anti-buckling fixture was performed between standard compression platens.

## Results

The shear strength results are detailed in Table 3, with the corresponding averages and standard deviations given in Tables 4 and 5.

**Table 4 - Cell means : Shear strength.**

| Material<br>Laboratory | 1    | 2    | 3    |
|------------------------|------|------|------|
| 1                      | 62.9 | 36.1 | 58.9 |
| 2                      | 72.2 | 31.9 | 60.5 |
| 3                      | 63.7 | 33.4 | 54.9 |
| 4                      | 69.9 | 31.3 | 56.7 |
| 5                      | 55.2 | 23.9 | 46.0 |
| 6                      | 70.5 | 35.3 | 59.6 |
| Global mean            | 65.7 | 32.0 | 56.1 |

**Table 5 - Standard deviations: Shear strength.**

| Material<br>Laboratory | 1    | 2   | 3   |
|------------------------|------|-----|-----|
| 1                      | 3.7  | 2.6 | 2.5 |
| 2                      | 6.4  | 3.6 | 3.2 |
| 3                      | 11.2 | 2.4 | 3.6 |
| 4                      | 15.8 | 5.5 | 2.2 |
| 5                      | 2.2  | 3.2 | 1.4 |
| 6                      | 9.2  | 1.4 | 1.5 |

In order to ascertain the criticality of the notch depths in achieving a correct through thickness shear strength value, the notch overlaps were plotted against the shear strength data for each specimen, and are shown in Figure 5(a)-(c). These highlight:

- Materials 1 and 3: significantly over-cut

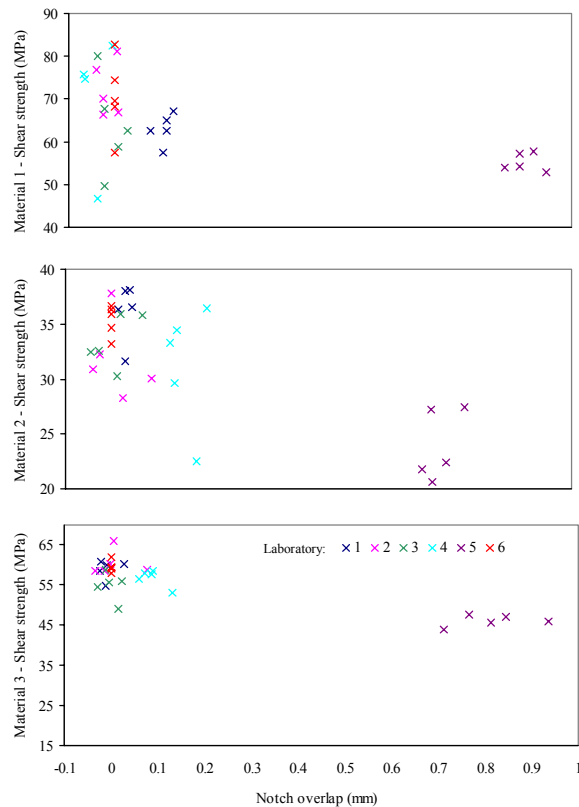


**Figure 5 - Shear strength vs. notch overlap data: (a)-(c) corresponding to materials 1-3 respectively.**

notch depths from Laboratory 5 resulted in the correct failure mode but with lower apparent shear strengths.

- Material 2: significantly over-cut notch depths from Laboratory 5 gave high shear strengths but with an incorrect mode of failure.
- Materials 2 and 3: plots show that if the specimen notch is either under or over-cut to within  $\pm 0.1$  mm of each other there is minimal difference in the results.

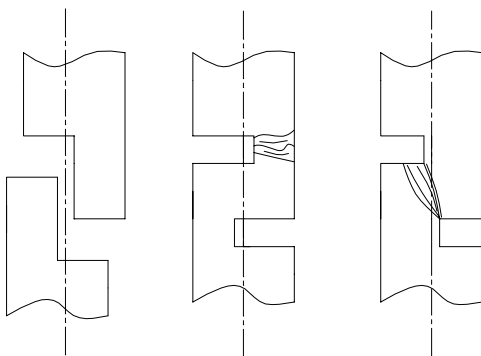


**Figure 6 - Failure modes: (a) Typical shear failure, (b)-(c) unacceptable failure modes.**

It was noted during testing that for material with similar through thickness shear compression strengths and a large notch overlap, an incorrect compressive failure occurred as illustrated in Figure 6(b). If, however the notches do not overlap and are machined short of each other, an artificially higher failure load can be achieved. This is shown in Figure 6(c), where a region of uncut material that can support a compressive load is present between the notches. Correct failure shown in Figure 6 (a) where the specimen fails uniformly, along the axis between the notch tips, into two equal parts.

### Statistical evaluation of results

Observations during testing and investigation of the results showed a clear irregularity:

- Laboratory 5: material 2 failures were invalid, failing across the notch, in addition lower mean results were obtained from the other two materials.

Specimen failures from all other laboratories were seen to be in through thickness shear, in accordance with the draft procedure, as shown in Figure 6(a) and Figure 7.



**Figure 7 - Typical specimen failures in materials 1-3 respectively.**

Due to the irregularities of the specimen manufacture and the erroneous results, the data from laboratory 5 was excluded from any further analysis.

Data from the remaining participants was then analysed in accordance with ISO 5725-2<sup>[1]</sup> using NPL in-house written software.

The data from the remaining 5 participants is shown graphically in Figure 8(a)-(c). These

show:

- Laboratory 4: potentially has individual outliers for materials 1 and 3.
- No laboratory: consistently produces higher or lower mean values from the global average.
- Material 1: shows a larger inter-laboratory scatter.
- Laboratory 4: generally exhibits larger intra-laboratory standard deviations than the other participants.

Using Mandel's statistics on the data, a graphical representation of the laboratory consistency can be shown. Mandel's  $h$  statistic, shown in Figure 9(a), is a measure of how the mean value for each laboratory and material differs from the mean value amongst all participating laboratories. The  $k$  statistic, shown in Figure 9(b), is the measure of standard deviation for each laboratory and material in relation to the sum of the variances for all the laboratories. The 1% and 5% significance levels are shown on the plots. If a value lies beyond these limits it needs to be inspected as it could be classified as a straggler or outlier. Using this method, we can verify that Laboratory 4 shows statistically greater scatter when compared to the other laboratories for materials 1 and 2.

Two differing statistical tests were then conducted to find any numerical outliers within the data:

Cochran's test measures within-laboratory variability against the sum of variances for that material in all participating laboratories.

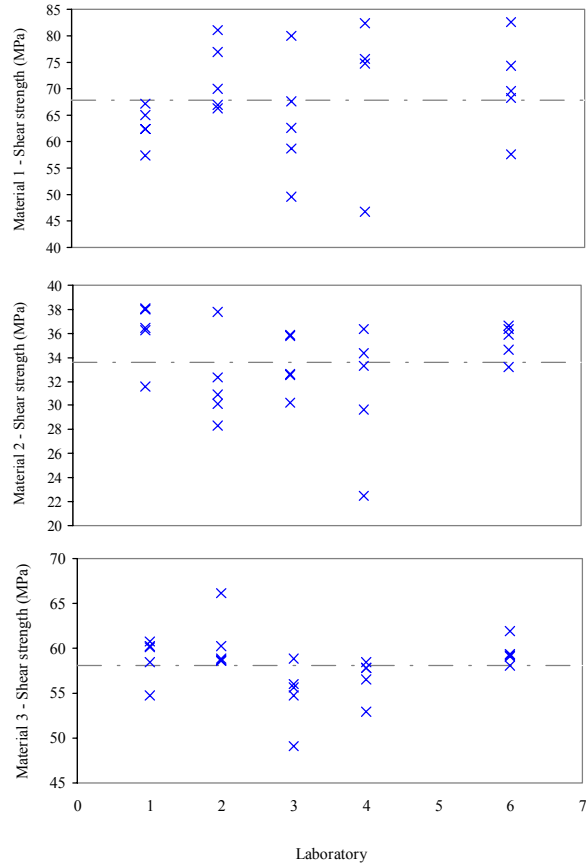
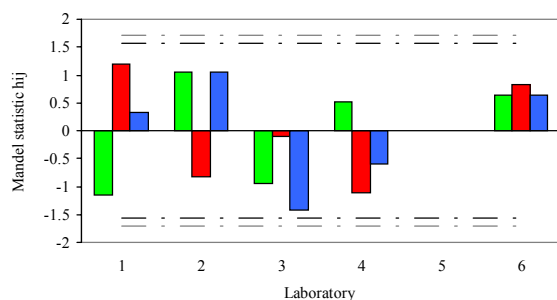


**Figure 8 - Data from double notch shear test: (a)-(c) corresponding to materials 1-3 respectively, grey line represents global mean.**

However this criterion tests only for high values and so is a one-sided test for outliers. Application of this test to the data indicated no stragglers or outliers.

Grubb's test measures the difference between the mean value from a laboratory against the average mean value of all the participating laboratories for a given material. Application of this test to the data also indicated no stragglers or outliers.

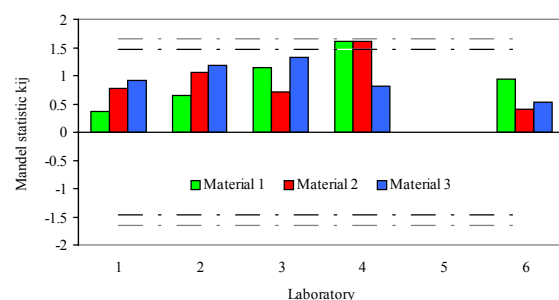


**Figure 9(a)-(b)- Mandel's  $h$  and  $k$  statistics for all included laboratories, grey and black lines represent the 1% and 5% significance levels respectively.**

**Table 6 - Repeatability and reproducibility limits and standard deviations.**

| Material | Repeatability Conditions |      | Reproducibility Conditions |      | Mean $\tau$<br>(MPa) |
|----------|--------------------------|------|----------------------------|------|----------------------|
|          | $S_r$                    | $r$  | $S_R$                      | $R$  |                      |
| 1        | 9.8                      | 27.3 | 9.8                        | 27.3 | 67.8                 |
| 2        | 3.4                      | 9.5  | 3.7                        | 10.4 | 33.6                 |
| 3        | 2.7                      | 7.5  | 3.3                        | 9.3  | 58.1                 |

The previous statistical tests focus on identifying “high” outlying results therefore the expected “low” outliers were not substantiated statistically for materials 1 and 2. Further investigation into the specimens in question showed that they had either not been machined square or had machining defects along one edge. However, similar defects had been noted on other specimens within the same batch, so the suspect data points have not been excluded from the analysis.

### Precision data

The repeatability and reproducibility of the double notch shear test method was then investigated, using the accepted data.

Repeatability,  $r$ , refers to tests that are conducted within the same test house using conditions that are as constant as possible:

- single operator,
- same equipment,
- tested in short time interval.

Reproducibility,  $R$ , refers to test that are preformed in a wide variety of conditions:

- different laboratories,
- different operators,
- different equipment.

Thus repeatability and reproducibility are two extremes, with repeatability measuring the minimum and reproducibility measuring the maximum variability in the precision of the results and therefore the test method.

Table 6 details the repeatability and reproducibility,  $r$  and  $R$ , of the specimen manufacture and testing; the repeatability and reproducibility standard deviations,  $s_r$  and  $s_R$  and the general mean. Table 7 represents this same data as a percentage of the mean shear strength,  $\tau$ .

### Concluding remarks

The data presented in this report suggests the following:

- The test method reproducibility and repeatability is both material and machining dependant, with the highest variability being from material 1. This material is both brittle and proved the most difficult to machine.
- The current procedure highlighted a problem with the machining of the specimen notch depths to the accuracy required. A recommendation can be made that the notch overlap tolerance be  $\pm 0.1$  mm and as close to the specimen centre line as possible, instead of requiring each notch to be exactly to the sample thickness centre line.
- The round-robin confirmed findings from previous exercises that the specimen manufacture/machining can have a marked effect on the failures loads and modes, and thus the repeatability and reproducibility of the results.
- The mean values obtained from this round-robin are comparable to other data obtained for this test method from similar materials <sup>[8]</sup>.

**Table 7 - Repeatability and reproducibility data expressed as percentage of the mean.**

| Material | Repeatability Conditions |         | Reproducibility Conditions |         |
|----------|--------------------------|---------|----------------------------|---------|
|          | $S_r$ (%)                | $r$ (%) | $S_R$ (%)                  | $R$ (%) |
| 1        | 14.4                     | 40.3    | 14.4                       | 40.3    |
| 2        | 10.1                     | 28.3    | 11.0                       | 31.0    |
| 3        | 4.6                      | 12.9    | 5.7                        | 16.0    |

## References

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- Fibreforce Composites
- INSYS Ltd
- Imperial College
- National Physical Laboratory
- Slingsby Aviation Ltd

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