A Standard Qualification Plan for Composite Material Systems

Reducing qualification costs in the composite materials industry

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Content of presentation

1. Introduction
2. Formulation of Standard Qualification Plan (SQP)
3. Round-robin validation of test methods
4. Results analysis procedure
5. Finalisation of SQP
6. Future work
1- Introduction

What is the Standard Qualification Plan?

Standard Qualification Plan (SQP): a set of composite material test standards that will meet the minimum common requirements necessary to allow:

- Quality control
- Initial material selection
- Preliminary design
Why Is a Qualification Plan Needed?

Costly to

.....qualify a product against different specifications

Material A

ASTM

ISO

BS

DIN
Why Is a Qualification Plan Needed?

Costly to

....qualify a product against different specifications

Material A

ASTM

ISO

BS

DIN
Why Is a Qualification Plan Needed?

Costly to

….introduce new materials because of qualification costs
Why Is a Qualification Plan Needed?

Difficult to

….find data for materials selection and preliminary design

Material A

Material B

Material C

LACK OF DATA

X
Feasibility

• SQP feasible due to recent availability of:
  – test panel manufacture standard (ISO 1268)
  – suite of harmonised test methods (mechanical, thermal and physical – see http://www.npl.co.uk/npl/cmmt/cog/index.html)
  – a data-sheet database standard (ISO 10350-2)

• New composite materials could be released with qualification data

• Previous consultation with industry indicated considerable support
Beneficiaries

• End-users/designers

• Suppliers of prepreg composite materials

• Certification bodies e.g. CAA, FAA etc.

• Test houses
Project Objectives

1. Develop a *Standard Qualification Plan (SQP)* aimed at significantly *reducing qualification costs*

2. Demonstrate to industry, the *suitability* and *robustness* of test methods proposed in the SQP

3. *Recommend* and *disseminate* to industry
2 Formulation Process

• Extensive industry consultation to agree content of plan
  – tests most commonly required/ performed
  – test standards being used
  – use of test data
  – importance of individual test methods and the need for their inclusion in the SQP

• Output - a draft report with an industrial feedback mechanism
Test Methods

UNCURED
- Mass per unit area
- Resin flow
- Fibre mass per unit area
- Percentage of volatile matter
- Resin and fibre volume fraction
- Glass transition temperature
- Analysis by DSC
- Density of fibre
- Density of resin
- Gel time

CURED
- Tension – unidirectional
- Tension - multidirectional
- Compression – unidirectional
- Compression – multidirectional
- Shear $\pm 45^\circ$ tension
- Shear strength – In-plane
- ILSS – Through-thickness
- Flexural
- Mode I fracture toughness
- Mode II fracture toughness
- Filled/open hole tension
- Filled/open hole compression
- Pin-bearing (plain, un-torqued)
- Bolted-joint bearing (torqued)
- Compression-after-impact
- Fatigue
- Creep
- Coefficient of thermal expansion
- Moisture uptake/conditioning
- Effect of water/moisture
- Effect of chemicals
- Effect of heat ageing
Questionnaire results

• **uncured properties** required for *materials selection* and *quality assurance* purposes

• **uncured property** test methods - *medium importance*, much of this data covered by material suppliers anyway.

• **cured properties** used mainly for materials *selection* and *design*

• **cured property** test methods - *high importance*, especially:
  – Tension
  – Compression
  – Shear
  – ILSS through-thickness
SQP/EQP format

• Draft SQP/EQP written in the style of an ISO standard
• Two parts
  – Part A – rationale and instructions
  – Part B – report sheets
• Based on format of
  – BS EN ISO 11403-1 – Acquisition and presentation of comparable multi-point data
  – ISO 10350-2 - Acquisition and presentation of comparable single-point data
• ISO test standards chosen as default
• Data included for several ISO test temperatures after dry and hot/wet conditioning
<table>
<thead>
<tr>
<th>Property</th>
<th>Symbol</th>
<th>Standard</th>
<th>Unit</th>
<th>Test Condition</th>
<th>Test conditions and supplementary instructions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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<td>-55°C</td>
<td>70°C</td>
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<tr>
<td>Fibre and resin volume fraction</td>
<td>$V_r$</td>
<td>ISO 14127 and ISO 1183</td>
<td>%</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$V_r$</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tension</td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\sigma_{011}$</td>
<td>Unidirectional BS EN ISO 527-5</td>
<td>MPa</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{022}$</td>
<td>Unidirectional BS EN ISO 527-5</td>
<td>MPa</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$E_{11}$</td>
<td>Unidirectional BS EN ISO 527-5</td>
<td>MPa</td>
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<td>3</td>
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<tr>
<td></td>
<td>$E_{22}$</td>
<td>Unidirectional BS EN ISO 527-5</td>
<td>MPa</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$\epsilon_{11}$</td>
<td>Unidirectional BS EN ISO 527-5</td>
<td>%</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$\epsilon_{22}$</td>
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<td>%</td>
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<td>3</td>
</tr>
<tr>
<td></td>
<td>$\nu_{12}$</td>
<td>Multidirectional BS EN ISO 527-4</td>
<td>%</td>
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<td>3</td>
</tr>
<tr>
<td></td>
<td>$\nu_{13}$</td>
<td>Multidirectional BS EN ISO 527-4</td>
<td>%</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$\nu_{23}$</td>
<td>Multidirectional BS EN ISO 527-4</td>
<td>%</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Compression</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
|          | $\sigma_{011}$ | ISO 14126, unidirectional, multidirectional | MPa | 1 | 3 | 1 | 1 | 3 | Refer to standard for specimen dimensions and test details. Type A, B$_1$, and B$_2$ specimens, testing by Method 1 (shear loading) or Method 2 (end loading)
|          | $\sigma_{022}$ | ISO 14126, unidirectional, multidirectional | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $E_{11}$ | ISO 14126, unidirectional, multidirectional | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $E_{22}$ | ISO 14126, unidirectional, multidirectional | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $\epsilon_{11}$ | ISO 14126, unidirectional, multidirectional | % | 1 | 3 | 1 | 1 | 3 | |
|          | $\epsilon_{22}$ | ISO 14126, unidirectional, multidirectional | % | 1 | 3 | 1 | 1 | 3 | |
| Shear ±45° tension |        |          |      |       |      |      |    |      |            |
|          | $\sigma_{011}$ | BS EN ISO 14129 | MPa | 1 | 3 | 1 | 1 | 1 | |
|          | $\sigma_{022}$ | BS EN ISO 14129 | MPa | 1 | 3 | 1 | 1 | 1 | |
|          | $\nu_{12}$ | BS EN ISO 14129 | % | 1 | 3 | 1 | 1 | 1 | |
|          | $\nu_{21}$ | BS EN ISO 14129 | % | 1 | 3 | 1 | 1 | 1 | |
| ILSS – through thickness |        |          |      |       |      |      |    |      |            |
|          | $\sigma_{011}$ | BS EN ISO 14130 | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $\sigma_{022}$ | BS EN ISO 14130 | MPa | 1 | 3 | 1 | 1 | 3 | |
| Flexural |        |          |      |       |      |      |    |      |            |
|          | $\sigma_{011}$ | BS EN ISO 14125 | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $\sigma_{022}$ | BS EN ISO 14125 | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $E_{11}$ | BS EN ISO 14125 | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $E_{22}$ | BS EN ISO 14125 | MPa | 1 | 3 | 1 | 1 | 3 | |
|          | $G_{12}$ | BS EN ISO 14125 | MPa | 1 | 3 | 1 | 1 | 3 | |

N.B. * - for 180°C cure resins only
Specimen Sampling

• Each property (per test environment) to be determined from a series of 30 tests

• 3 material batches
  – 2 panels per batch
    – 5 specimens per panel

• MIL-HDBK-17 recommends a min. of 30 specimens taken from at least 5 batches
Specimen Sampling

- BATCH 1
  - PANEL 1: 5 SPECIMENS
  - PANEL 2: 5 SPECIMENS

- BATCH 2
  - PANEL 3: 5 SPECIMENS
  - PANEL 4: 5 SPECIMENS

- BATCH 3
  - PANEL 5: 5 SPECIMENS
  - PANEL 6: 5 SPECIMENS
Presentation of results

• Results presented in report sheets of Part B
  – mean and standard deviations of 30 tests
  – individual results provided to allow further data analysis
  – procedure provided for generation of A- and B- basis design allowables – based on methods of MIL-HDBK-17

• Certain data values recommended to be normalised with respect to a nominal $V_f$ – procedure provided

• Normalise all mechanical stiffness and strength properties except:
  – $90^\circ$ (transverse) tension (UD laminates)
  – $90^\circ$ compression (UD laminates)
  – interlaminar shear
  – in-plane shear
  – short beam strength
  – bearing
  – strain energy release rates
  – Poisson’s ratio
3 Round-robin validation of test methods

- Round-robin (RR) conducted on 6 test methods
- Tests selected for RR dependent on importance of data, likelihood of error in testing, availability of previous data
- Analysis of results to ISO 5725
- Organisations were encouraged to assess at an early stage the use of the SQP in their operations
## Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Supplier</th>
<th>Description</th>
<th>Fibre type</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Hexcel</td>
<td>913 Carbon-T300J-5-35%</td>
<td>T300J (12k)</td>
</tr>
<tr>
<td>2</td>
<td>SP Systems</td>
<td>SE84LV/HSC/300/300/37±3%</td>
<td>T700 (24k)</td>
</tr>
</tbody>
</table>

*N.B. Both materials donated by industry*

*Typical of Aerospace/Formula 1 automotive applications*
Panel/specimen preparation

• Unidirectional specimens machined from 1 and 2 mm thick panels

• Test panels manufactured according to
  – ISO 1268 Part 4 – Preparation of fibre-reinforced, resin bonded, low-pressure, laminated plates or panels for test purposes

• Specimens extracted following
  – ISO 2818 – Preparation of test specimens by machining

• Additional machining guidance
  – Measurement Good Practice Guide - Machining of Composites and Specimen Preparation (NPL GPG No. 38)
## Test Methods

<table>
<thead>
<tr>
<th>Test</th>
<th>Standard</th>
<th>Properties measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension</td>
<td>BS EN ISO 527-1 and -5</td>
<td>$\sigma_{mt11}$, $E_{t11}$, $\nu_{12}$</td>
</tr>
<tr>
<td>Compression</td>
<td>BS EN ISO 14126</td>
<td>$\sigma_{mc11}$, $E_{c11}$</td>
</tr>
<tr>
<td>Flexure</td>
<td>BS EN ISO 14125</td>
<td>$\sigma_{mf11}$, $E_{f11}$</td>
</tr>
<tr>
<td>Interlaminar shear (ILSS)</td>
<td>BS EN ISO 14130</td>
<td>$\tau_{m1}$</td>
</tr>
<tr>
<td>DMA</td>
<td>ISO/CD 6721-11</td>
<td>$T_g$, $T_{onset}$, $T_{loss}$, $T_{tandelta}$</td>
</tr>
</tbody>
</table>

**Mechanical tests – 6 specimens, DMA – 3 specimens**

**Specimen preparation undertaken by NPL**
### Tensile tests to BS EN ISO 527-5

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of sites</td>
<td>8</td>
</tr>
<tr>
<td>Test speeds used (mm/min)</td>
<td>2 used by all</td>
</tr>
<tr>
<td>Load cell capacities (kN)</td>
<td>100, 250</td>
</tr>
<tr>
<td>Method of deflection measurement</td>
<td>• Biaxial strain gauge</td>
</tr>
<tr>
<td></td>
<td>• Dynamic serial gauge</td>
</tr>
<tr>
<td></td>
<td>• Biaxial extensometer</td>
</tr>
<tr>
<td></td>
<td>• Longitudinal and transverse extensometers</td>
</tr>
<tr>
<td></td>
<td>• Crosshead deflection</td>
</tr>
</tbody>
</table>

![Image of tensile test setup]

250 mm

15 mm

50 mm

Thickness = ~ 1 mm

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## Compression tests to BS EN ISO 14126

<table>
<thead>
<tr>
<th>Number of sites</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test speeds used (mm/min)</td>
<td>1 and 1.27</td>
</tr>
<tr>
<td>Load cell capacities (kN)</td>
<td>100, 200, 250</td>
</tr>
</tbody>
</table>
| Method of deflection measurement | • Biaxial strain gauges  
• Extensometer  
• Crosshead deflection |
| Loading jig | • Celanese  
• In-house end loading blocks |

### Diagram

- Material length: 110 mm
- Tab length: 10 mm
### Flexure tests to BS EN ISO 14125

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of sites</td>
<td>9</td>
</tr>
</tbody>
</table>
| Test speeds used (mm/min) | 1, 2, 5, 5.21, 6.6, 6.75, 7  
(3 sites used different test speeds for each material) |
| Load cell capacities (kN) | 1, 5, 10, 20, 50, 100 |
| Span (3-point loading for all sites) | • 7 sites used different span for each material  
• 2 sites used one span for both materials |
| Method of deflection measurement | • LVDT  
• Crosshead deflection |

### ILSS tests to BS EN ISO 14130

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of sites</td>
<td>6</td>
</tr>
<tr>
<td>Test speeds used (mm/min)</td>
<td>1 used by all</td>
</tr>
<tr>
<td>Load cell capacities (kN)</td>
<td>5, 100</td>
</tr>
<tr>
<td>Span</td>
<td>All sites used different span for each material</td>
</tr>
</tbody>
</table>
# DMA tests to ISO/CD 6721-11

<table>
<thead>
<tr>
<th>Number of sites</th>
<th>5</th>
</tr>
</thead>
</table>
| **Test mode**   | • 1 site used a 3-point bend configuration  
                  • 3 sites used a single cantilever bend configuration  
                  • 1 site used single and dual cantilever configurations |
| **Test span (range)** | • 20 mm for 3-point bend  
                        • 10, 15, 17.5 mm for single cantilever bend  
                        • 20 mm for dual cantilever bend |
| **Frequency**   | 1 Hz used by all |
| **Temperature range (°C)** | 25-200, 25-250, 25-300 |
| **Heating rate (°C/min)** | 3 |
Participants

• Advanced Composites Group
• Motor Industry Research Association (MIRA)
• Ford Motor Company
• Slingsby Aviation
• Aerostructures Hamble
• Composites Testing Laboratory
• Bureau Veritas
• Gearing Scientific
• Triton Technology
• Perkin Elmer Thermal Analysis Solutions
• NPL
## Properties measured

<table>
<thead>
<tr>
<th>Site</th>
<th>Tension</th>
<th>Compression</th>
<th>Flexure</th>
<th>ILSS</th>
<th>DMA</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\sigma_{M_{t11}}$</td>
<td>$E_{t11}$</td>
<td>$\nu_{12}$</td>
<td>$\sigma_{M_{c11}}$</td>
<td>$E_{c11}$</td>
</tr>
<tr>
<td>1</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>2</td>
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<td></td>
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<tr>
<td>3</td>
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<tr>
<td>Total</td>
<td>8</td>
<td>8</td>
<td>4</td>
<td>5</td>
<td>3</td>
</tr>
</tbody>
</table>
4 Analysis of results

• **ISO 5725-2** - Accuracy (trueness and precision) of measurement methods and results -- Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

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I. Initial check of “as-received” data

II. Mandel's $h$ and $k$ statistics

III. Cochran and Grubb tests for outliers and stragglers

IV. Calculation of repeatability and reproducibility
I - Check of “as-received” data

Tensile strength

Tensile modulus

Compression strength
II – Mandel’s h and k consistency statistics

Tensile modulus - example of normal pattern

Between site consistency, $h_{ij}$

Within site consistency, $k_{ij}$

Material 1

Material 2
II – Mandel’s h and k consistency statistics

Flexural modulus - example of abnormal pattern

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III – Cochran and Grubb tests

• Cochran test – site variances

• Grubb test – site means and outlying observations

• Following ISO 5725:
  – Outliers discarded
  – Stragglers retained

• On basis of statistical tests some further data discarded
Tension results

- Explosive fracture
- Various deflection measurement methods used
- Good repeatability and reproducibility after removal of erroneous data
Compression results

- Acceptable failure modes achieved
- Only 1 site checked for bending
- High values of repeatability and reproducibility for strength
- Few sites able to undertake tests
Flexure results

- Acceptable failure modes achieved
- Various deflection measurement methods used
- Various test speeds used
- Systematic errors in measurement observed for 2 sites
- Good repeatability and reproducibility after removal of erroneous data
ILSS results

- Failure modes unacceptable for all sites and both materials

- Data analysed as purpose of this round-robin was not to generate precision data

- Good repeatability and reproducibility after removal of erroneous data
DMA results

- Double peaks reported on $T_{\text{tan}\delta}$ plots for material 1 – not fully cured
- Some difficulties specifying onset and loss modulus peaks
- Very low repeatability
- Higher reproducibility due to:
  - Deficiencies in temperature measurement
  - Various methods for temperature calibration
IV – Repeatability and reproducibility

Repeatability CoV - Material 1
Repeatability CoV - Material 2
Reproducibility CoV - Material 1
Reproducibility CoV - Material 2

σ_{M11}  E_{t11}  ν_{12}  σ_{M11}  E_{c11}  σ_{Mf11}  E_{f11}  τ_{M1}  T_g  T_{onset}  T_{loss}  T_{tandelta}

TENSION  COMPRESSION  FLEXURE  ILSS  DMA
Conclusions

- Tensile, flexure, ILSS and compression modulus tests showed good repeatability and reproducibility values.

- Compression strength showed considerable within and between site variability – alignment crucial (essential to check for bending).

- Further development and guidance needed for compression testing aspects such as strain measurement, end-tab design and testing of thick sections.
Conclusions

• Improvements required in DMA temperature measurement and calibration
  – NPL has developed 3 types of calibration specimen – one of which will be used in a DMA calibration standard (ISO/CD 6721-12)

• Use of accurate, calibrated equipment essential for measuring:
  – Load
  – Displacement
  – Specimen dimensions (to accuracy required by standard)

• Displacement/strain should where possible be measured using:
  – Clip gauge extensometers
  – Strain gauges
  – LVDT/dial gauge indicators

• At the very least the crosshead displacement should only be used in conjunction with a machine compliance correction
5 Finalisation of SQP

- Finalisation of SQP as a *Good Practice Guide* based on feedback from draft and round-robin exercise

- Used as a *pre-cursor for standardisation*

- *Promoted widely* to the composites industry

- Assessment of current *status of material databases*
6 Future work

- Further effort required for development toward standardisation
- Further collaboration with CAA
- Collaboration with similar international initiatives (e.g. FAA)
- Development of SQPs for other materials/sectors
- Additional dissemination to promote and encourage adoption