A Standard Qualification Plan for Composite Material Systems

Reducing qualification costs in the composite materials industry

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COMPTEST 2004, 21-23 September
University of Bristol

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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?

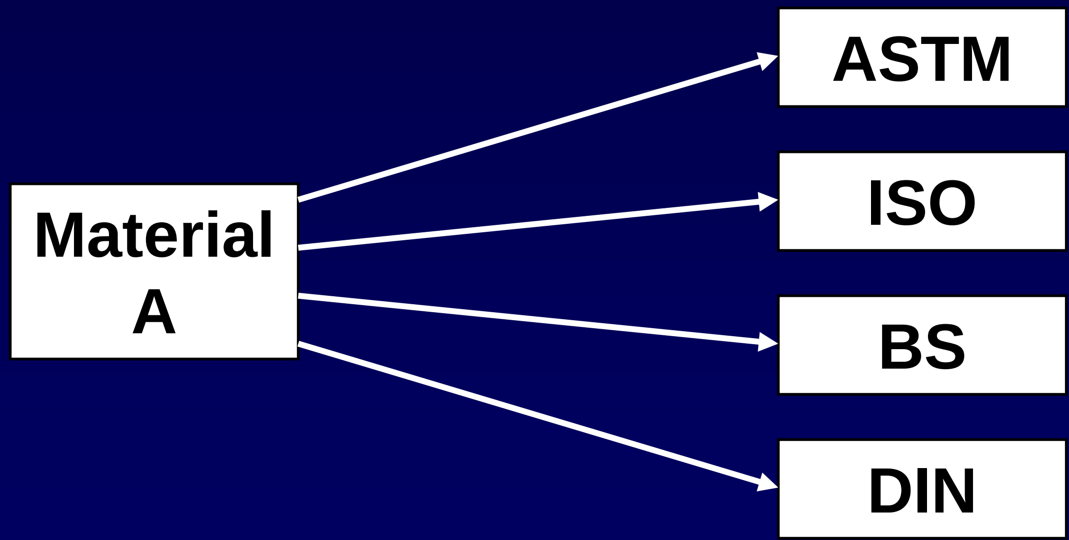
Standard Qualification Plan (SQP): a *set of composite material test standards* that will meet the minimum common requirements necessary to allow:

- Quality control
- Initial material selection
- Preliminary design

Why Is a Qualification Plan Needed?

Costly to

….qualify a product against different specifications



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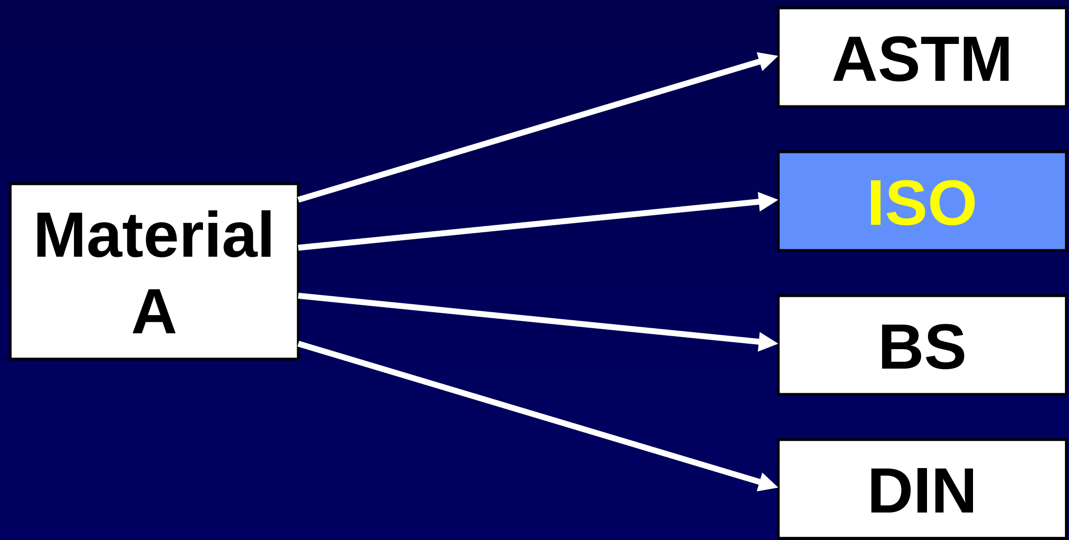
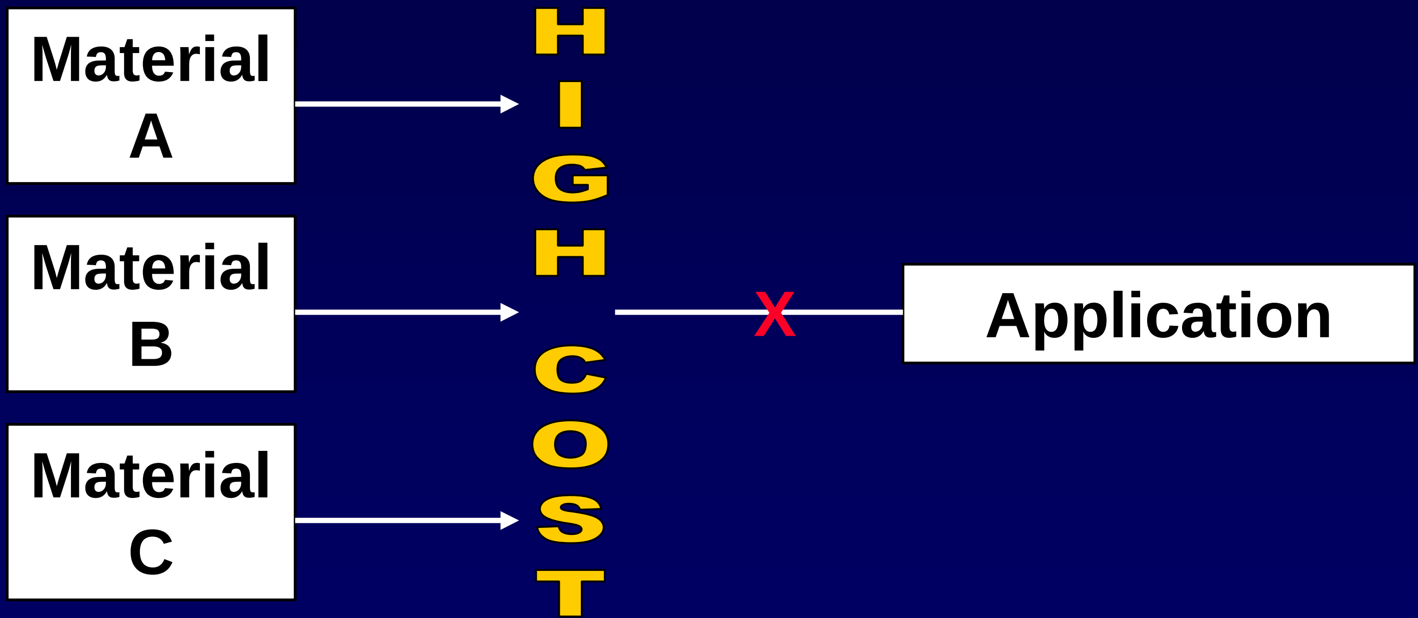


Why Is a Qualification Plan Needed?

Costly to

….introduce new materials because of qualification costs



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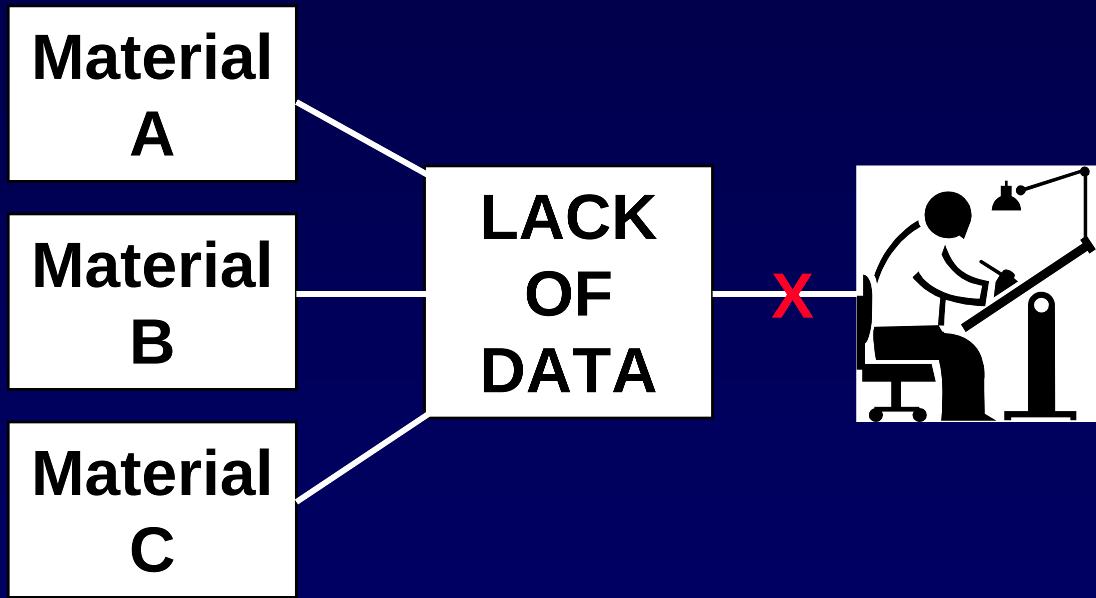
Why Is a Qualification Plan Needed?

Difficult to

….find data for materials selection and preliminary design



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
- Gell time

CURED

- Tension – unidirectional
- Tension - multidirectional
- Compression – unidirectional
- Compression – multidirectional
- Shear ± 45° 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

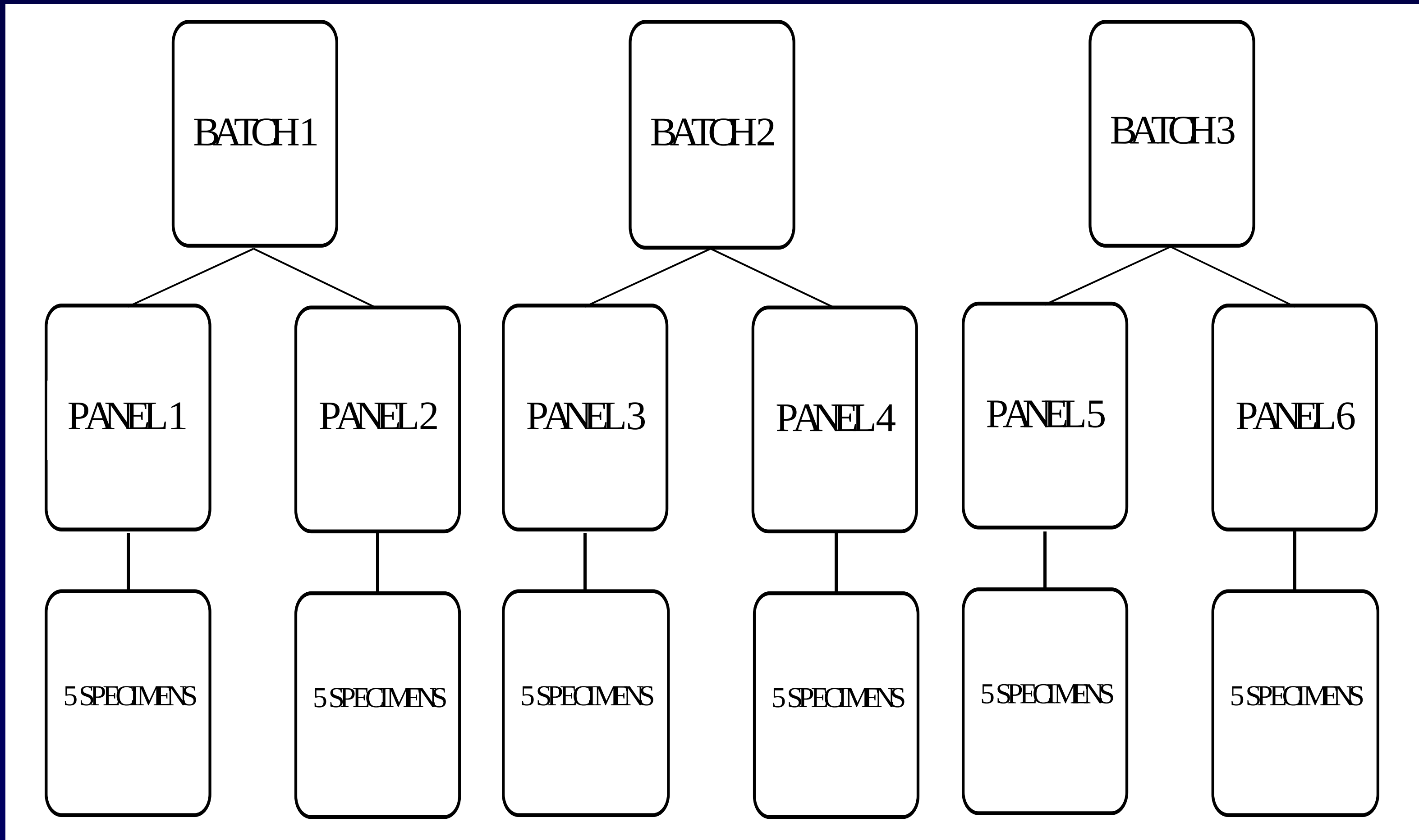
	Property	Symbol	Standard	Unit	Test Condition: DRY -55°C	DRY RT	DRY 70°C	DRY 125°C*	70°C/85% RH RT	70°C/85% RH 70°C	Test conditions and supplementary instructions
14	Fibre and resin volume fraction	V_f	ISO 14127 and ISO 1183	%		3					ISO 1183 for density
		V_r				3					
15	Tension	σ_{Mt11}	Unidirectional BS EN ISO 527-5 Multidirectional BS EN ISO 527-4	MPa	1	3	1			1	Refer to standard for specimen dimensions and test details. () – not required for balanced fabrics
		σ_{Mt22}		MPa	1	1	(1)			(1)	
		E_{t11}		GPa	1	3	1			1	
		E_{t22}		GPa	1	1	(1)			(1)	
		ε_{Mt11}		%	1	3	1			1	
		ε_{Mt22}		%	1	1	(1)			(1)	
		ν_{12}				1					
		ν_{13}				1					
		ν_{21}				1					
		ν_{23}				1					
16	Compression	σ_{Mc11}	ISO 14126 unidirectional, multidirectional	MPa	1	3	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) () – not required for balanced fabrics
		σ_{Mc22}		MPa		1	(1)			1	
		E_{c11}		GPa	1	3	1			3	
		E_{c22}		GPa		1	(1)			1	
		ε_{Mc11}		%	1	3	1			3	
		ε_{Mc22}		%		1	(1)			1	
17	Shear ±45° tension	τ_{M12}	BS EN ISO 14129	MPa	1	3	1	1	1	1	
		γ_{M12}		%	1	3	1	1	1	1	
		G_{12}		GPa	1	3	1	1	1	1	
18	ILSS – through thickness	τ_{M1}	BS EN ISO 14130	MPa	1	3	1	3	1	3	
		τ_{M2}		MPa		1	1				
19	Flexural	σ_{Mf11}	BS EN ISO 14125	MPa		3					Refer to standard for specimen dimensions and test details. Provide details of which method used: Method A (3 point flexure) and Method B (4 point flexure)
		σ_{Mf22}		MPa		3					
		E_{f11}		MPa		3					
		E_{f22}		MPa		3					
		G_{13}		GPa		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



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° (transverse) tension (UD laminates)
  - 90° 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

Material	Supplier	Description	Fibre type
1	Hexcel	913 Carbon-T300J-5-35%	T300J (12k)
2	SP Systems	SE84LV/HSC/300/300/37±3%	T700 (24k)

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

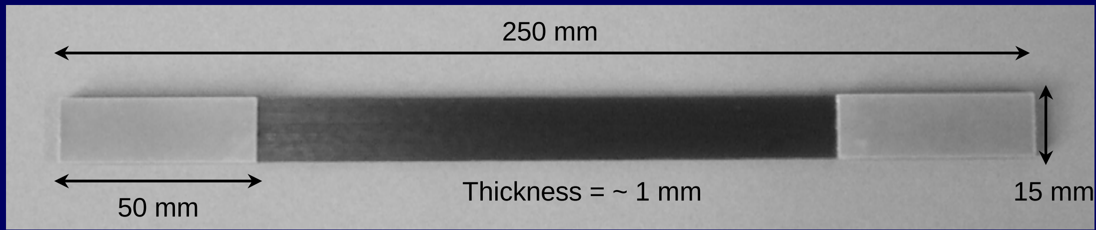
Test	Standard	Properties measured
Tension	BS EN ISO 527-1 and -5	σ_{mt11}, E_{t11}, ν_{12}
Compression	BS EN ISO 14126	σ_{mc11}, E_{c11}
Flexure	BS EN ISO 14125	σ_{mf11}, E_{f11}
Interlaminar shear (ILSS)	BS EN ISO 14130	τ_{m1}
DMA	ISO/CD 6721-11	T_g, T_{onset}, T_{loss}, $T_{tandelta}$

Mechanical tests – 6 specimens, DMA – 3 specimens

Specimen preparation undertaken by NPL

Tensile tests to BS EN ISO 527-5

Number of sites	8
Test speeds used (mm/min)	2 used by all
Load cell capacities (kN)	100, 250
Method of deflection measurement	• Biaxial strain gauge • Dynamic serial gauge • Biaxial extensometer • Longitudinal and transverse extensometers • Crosshead deflection



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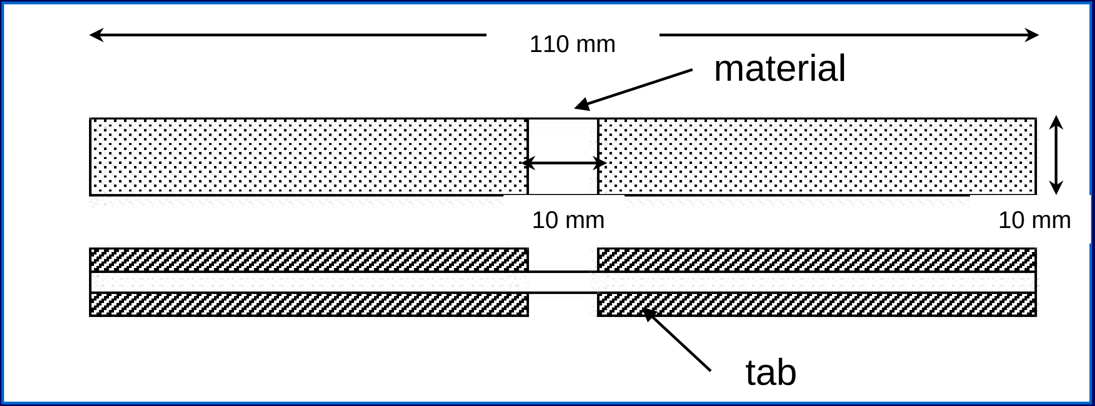
Compression tests to BS EN ISO 14126

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



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Flexure tests to BS EN ISO 14125

Number of sites	9
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

Number of sites	6
Test speeds used (mm/min)	1 used by all
Load cell capacities (kN)	5, 100
Span	All sites used different span for each material

DMA tests to ISO/CD 6721-11

Number of sites	5
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

Site	Tension			Compression		Flexure		ILSS	DMA
	σ_{Mt11}	E_{t11}	ν_{12}	σ_{Mc11}	E_{c11}	σ_{Mf11}	E_{f11}	τ_{M1}	
1	✔	✔	✔	✔	✔	✔	✔	✔	
2	✔	✔				✔	✔		
3	✔	✔	✔	✔	✔	✔	✔	✔	✔
4	✔	✔				✔	✔		
5	✔	✔	✔	✔	✔	✔	✔	✔	✔
6	✔	✔		✔		✔	✔		
7	✔	✔		✔		✔	✔	✔	
8						✔		✔	
9	✔	✔	✔			✔	✔	✔	
10									✔
11									✔
12									✔
Total	8	8	4	5	3	9	8	6	5

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**

Initial check of “as-received” data

I

Mandel’s *h* and *k* statistics

II

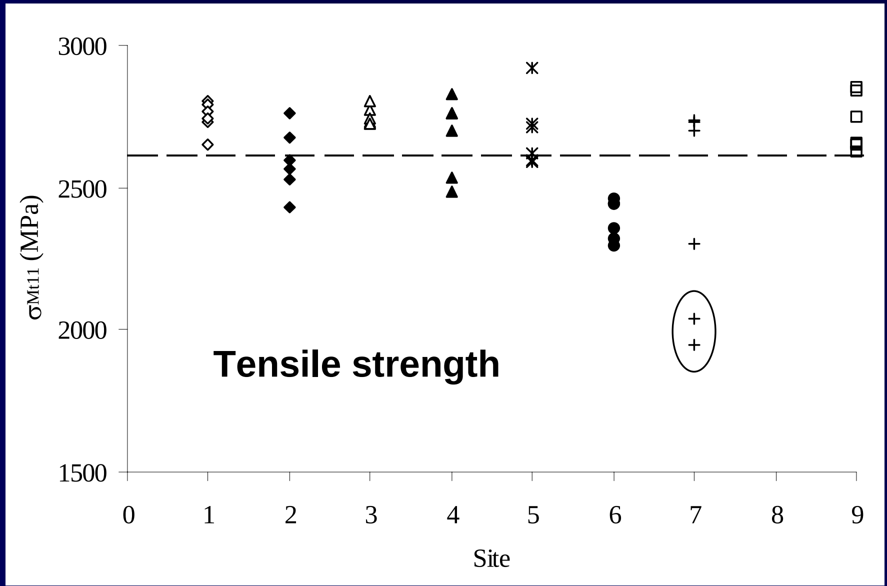
Cochran and Grubb tests for outliers and stragglers

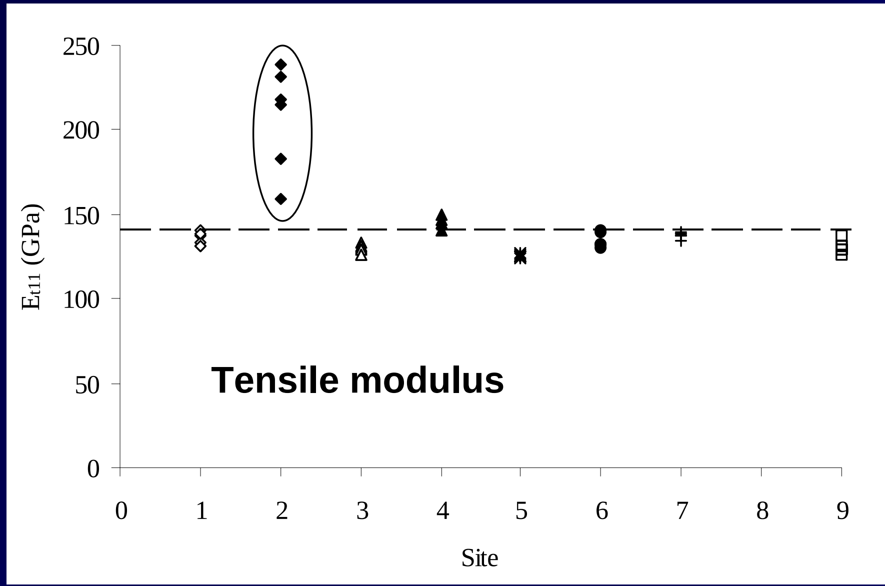
III

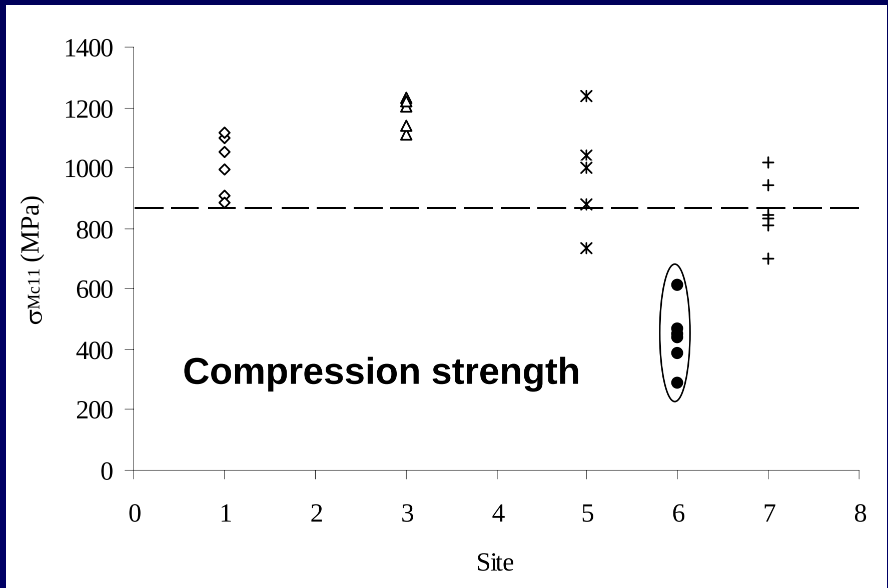
Calculation of repeatability and reproducibility

IV

I - Check of "as-received" data







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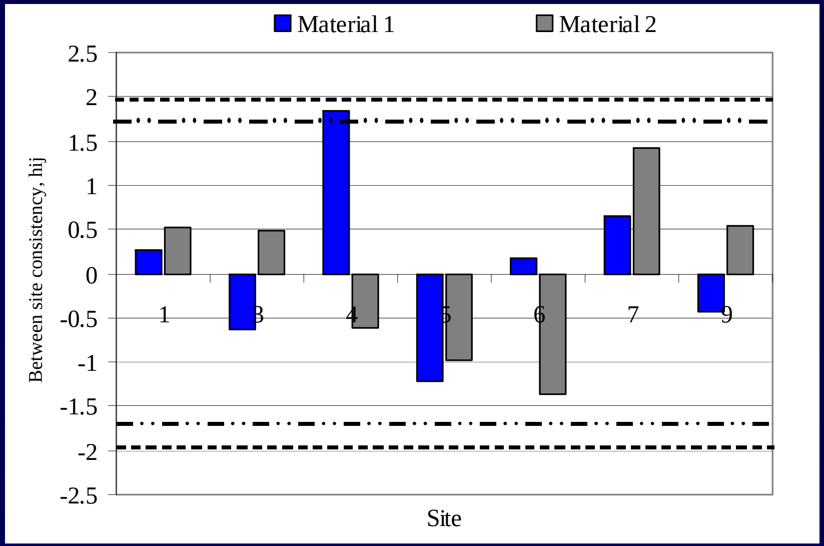
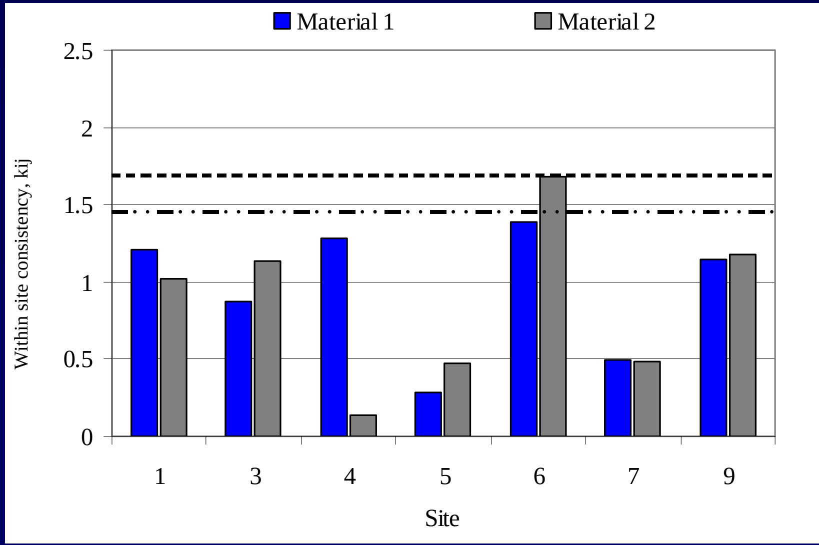
II – Mandel’s h and k consistency statistics



Tensile modulus - example of normal pattern



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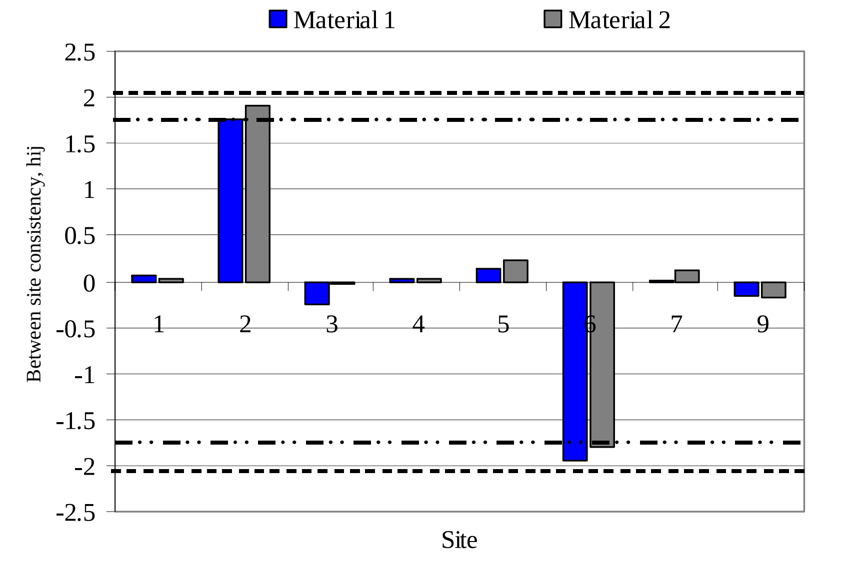
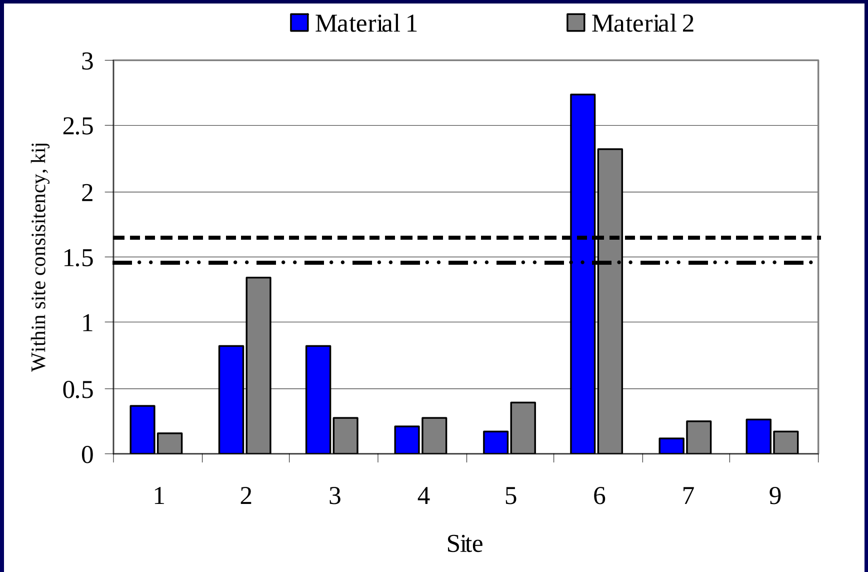
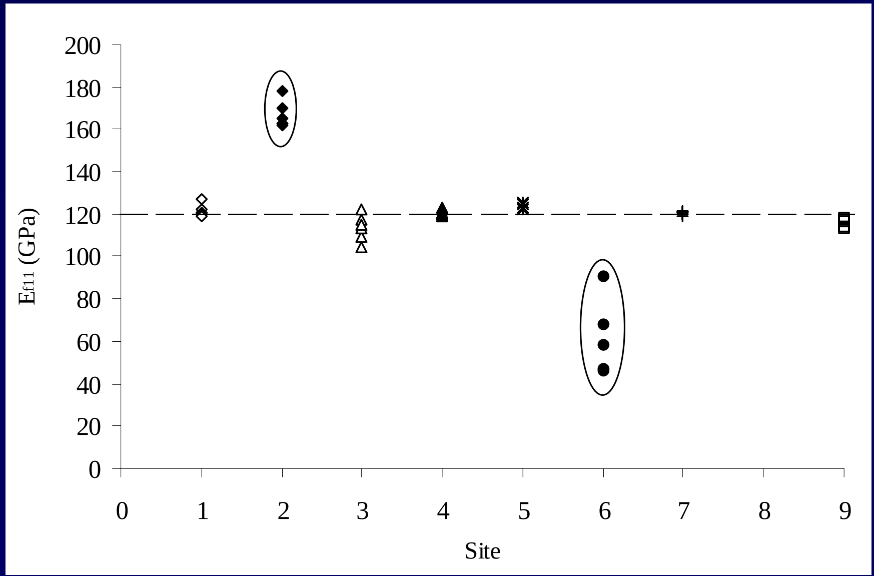


Flexural modulus - example of abnormal pattern





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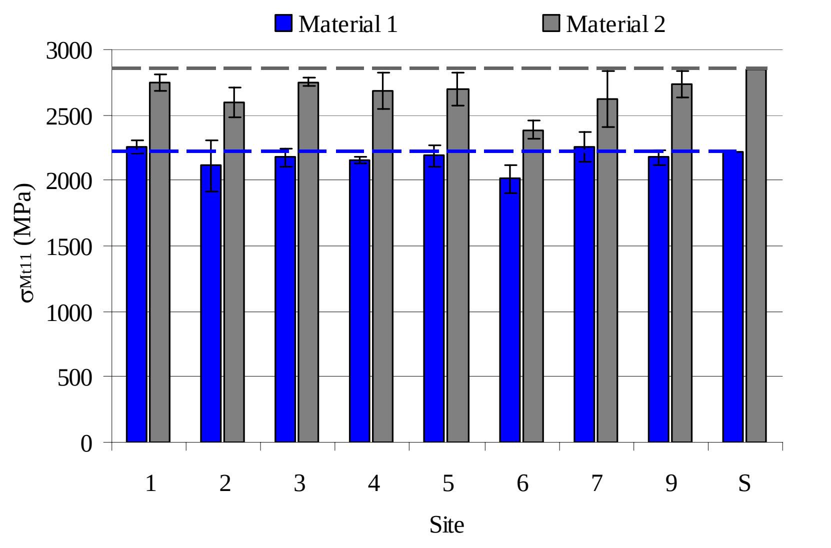
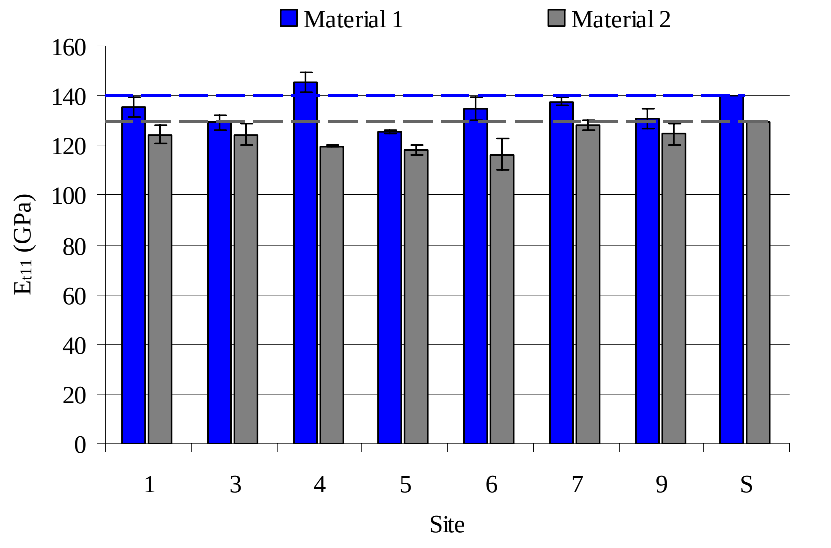
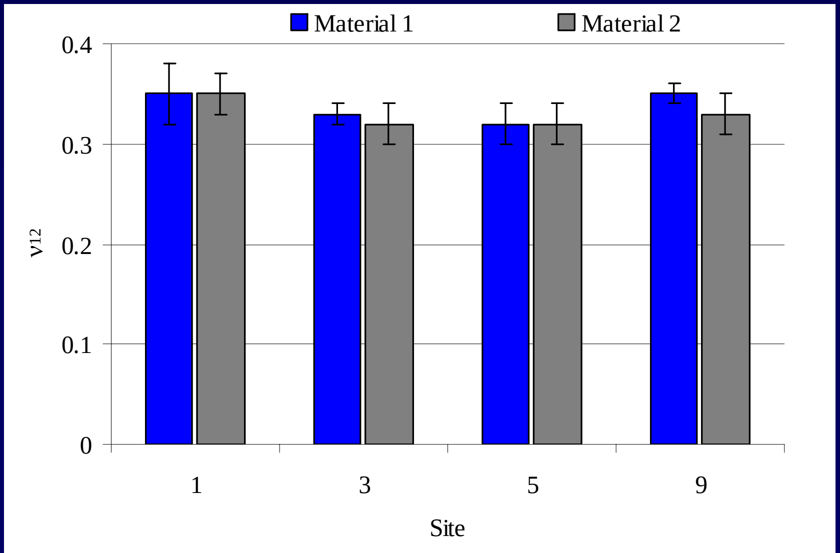
III – Cochran and Grubb tests

- 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

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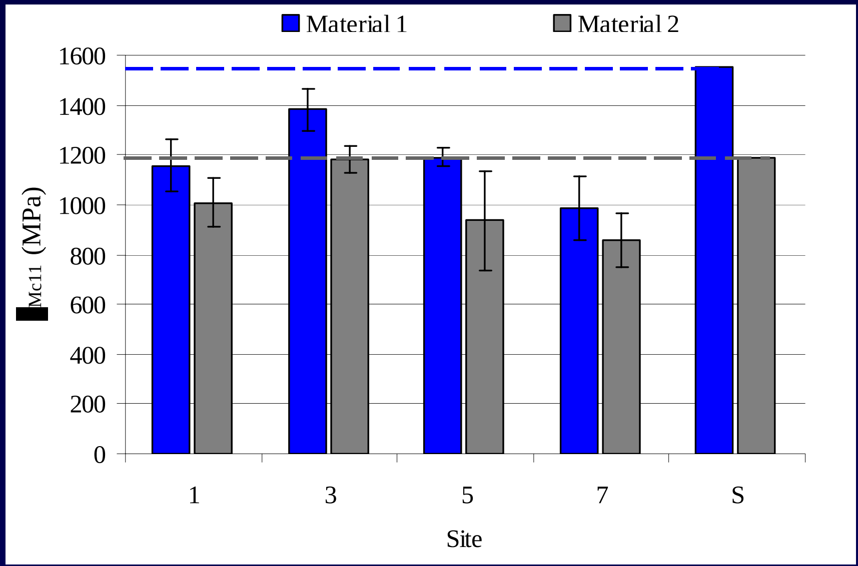
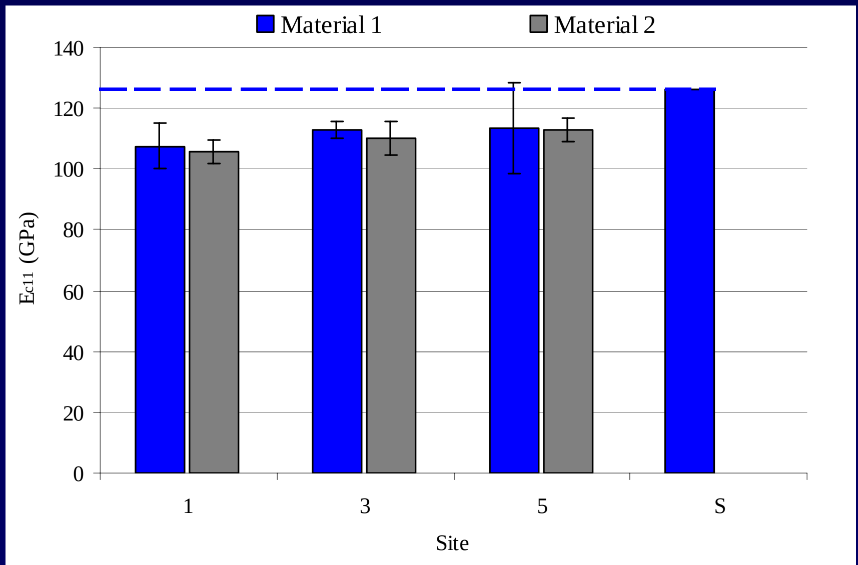
Compression results





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

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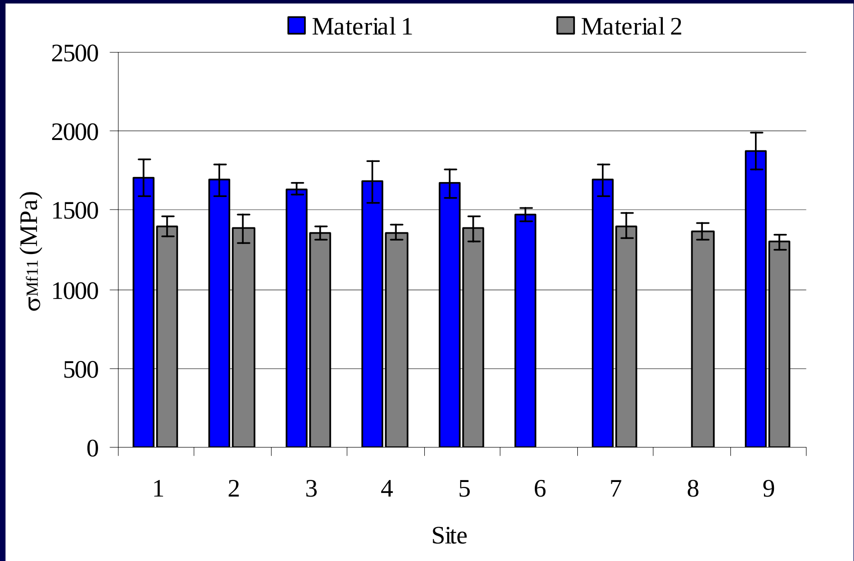
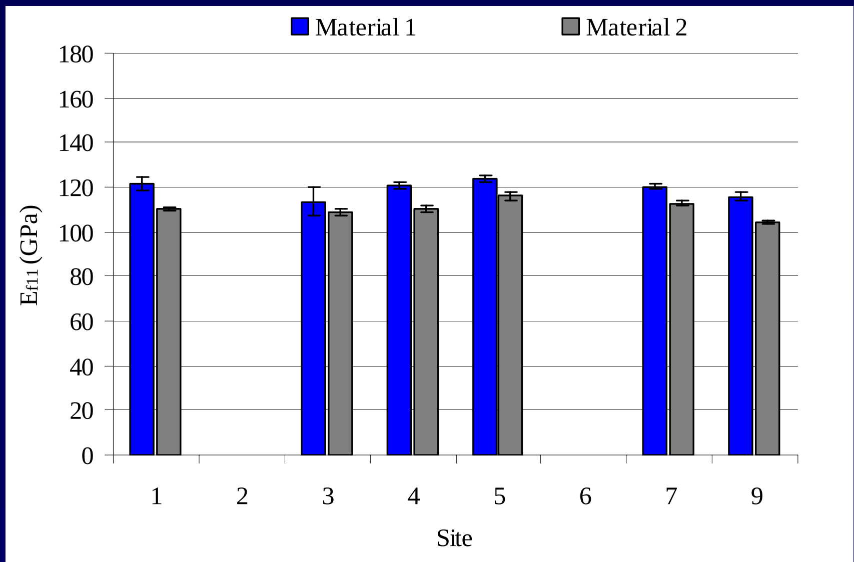
Flexure results





- 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

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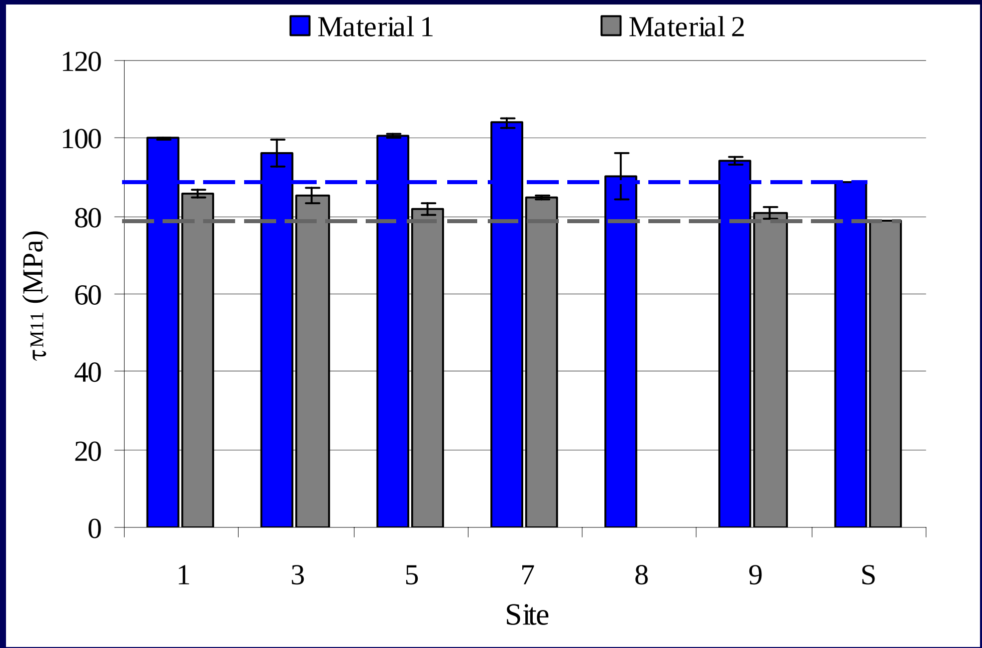
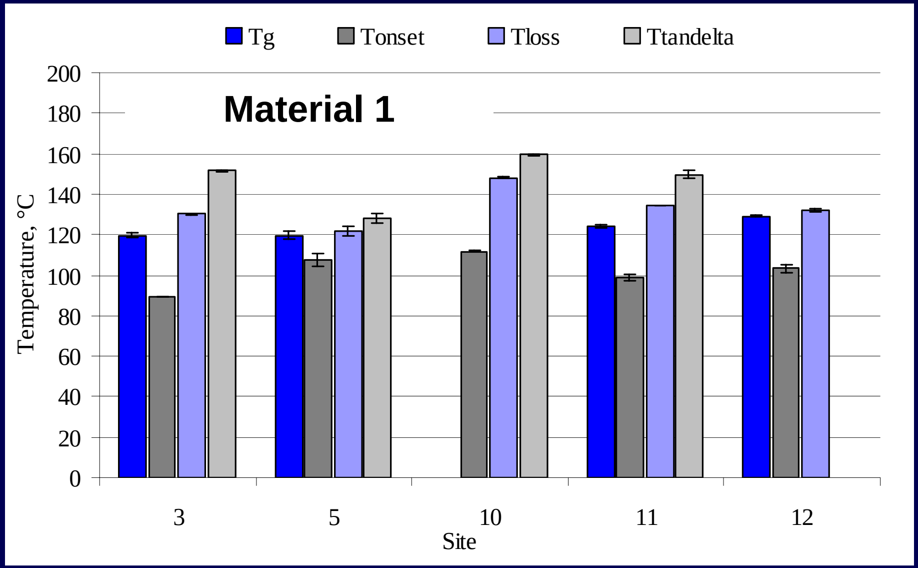
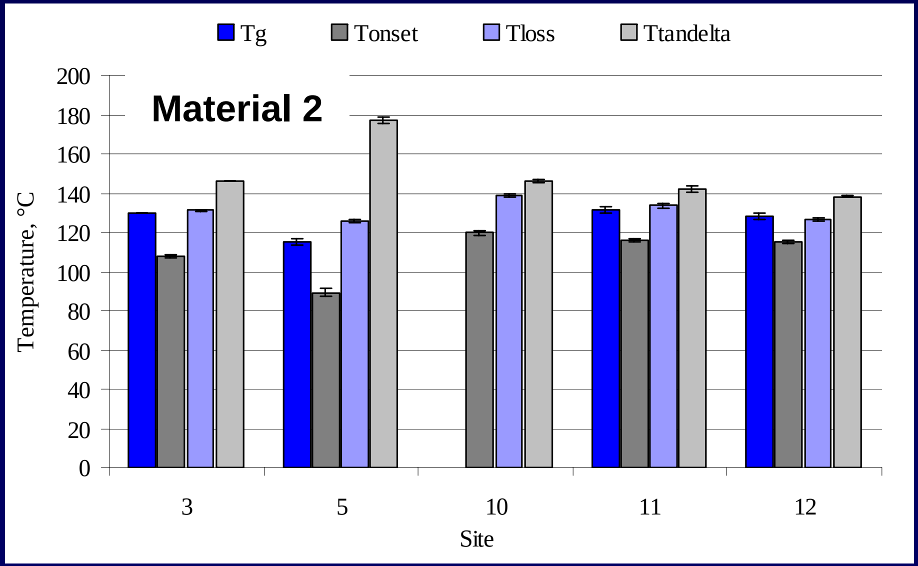
ILSS results



- 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

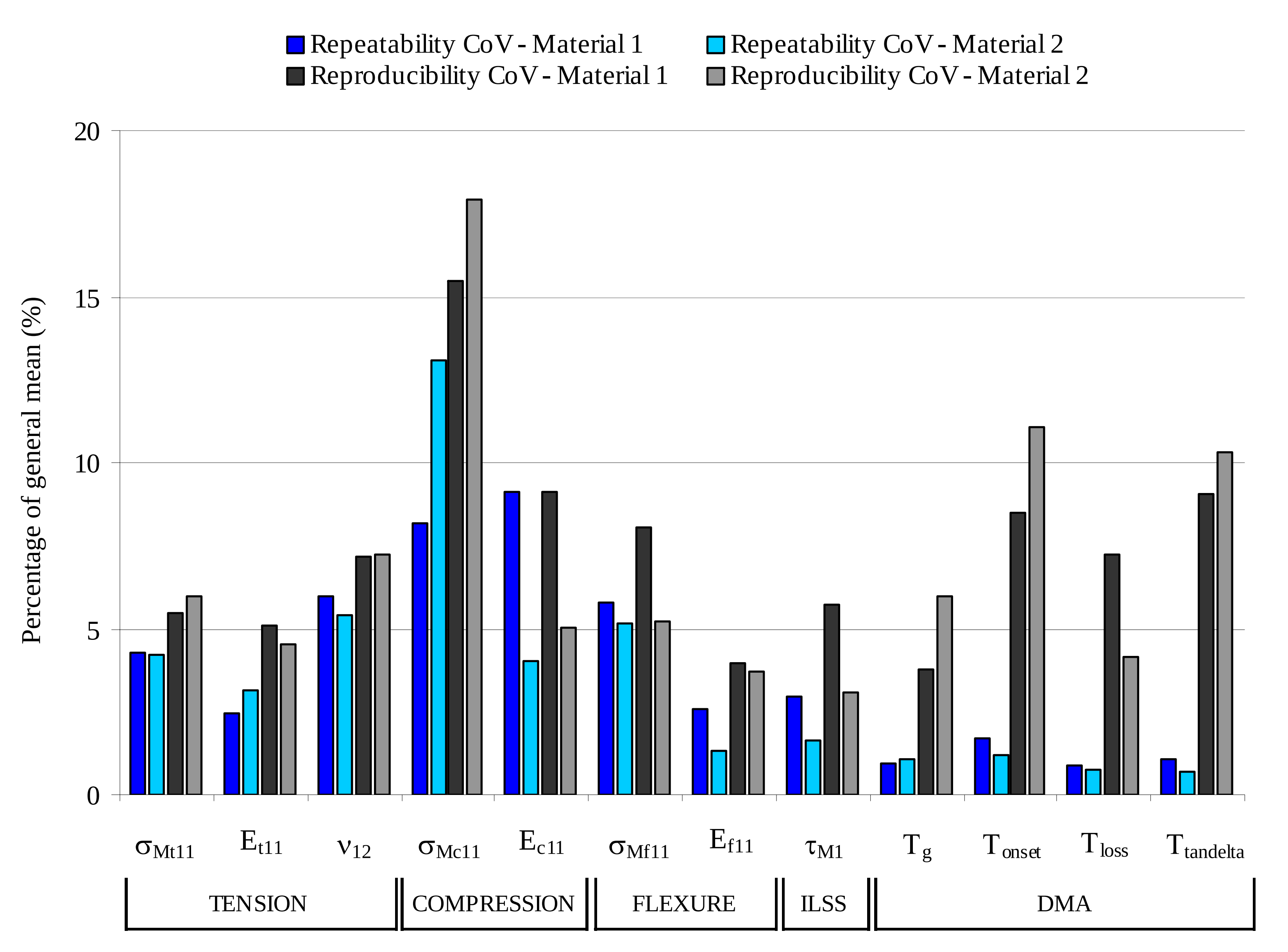
DMA results





- Double peaks reported on $T_{tandelta}$ 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



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

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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