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ASSESSING THE ROBUSTNESS OF UNCERTAINTY DETERMINATION FOR IN-PLANE PROPERTY DATASETS FOR FIBRE-REINFORCED POLYMER (FRP) COMPOSITES WITH DIRECT COMPARISON TO B-BASIS STATISTICAL ANALYSIS

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Assessing the Robustness of Uncertainty Determination for In-Plane Property Datasets for Fibre-reinforced Polymer (FRP) Composites with Direct Comparison to B-Basis Statistical Analysis

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CONTENTS

GLOSSARY/ABBREVIATIONS

A	A-basis value
B	B-basis value
k	Nonparametric B-basis factors for small sample sizes
k_b	One sided tolerance limit factor (Normal distribution)
n	Number of tests in a series
S	Standard deviation of a series of tests
U	Expanded uncertainty
V_b	One sided tolerance limit factor (Weibull distribution)
$\bar{x}$	Mean of a series of tests
$x_{(r)}$	r^{th} largest data value in sample
$x_{(1)}$	Smallest data value in a sample
σ_f	Flexural stress
$\hat{\alpha}$	Scale parameter of Weibull distribution
$\hat{\beta}$	Shape parameter of Weibull distribution

1 INTRODUCTION

The accurate knowledge of the properties of composite materials are critical for the design of structures and components used in a wide range of industries. They possess high specific strength and stiffness properties and are often preferable for component design, in comparison to materials such as steel, as they allow for similar material properties at a significant weight reduction. When designing a component and generating design allowables for a material property, it is critical to have confidence in the material property data so that a material can be selected which satisfies the design requirements whilst avoiding design conservatism and maintaining safety and confidence for critical infrastructure.

To provide confidence in the performance of a material, a 'weakest link' approach is often used when determining the design allowable value that is used in the modelling and design stages. The 'weakest link' theory suggests that a component will fail at the weakest point, often initiated at the site of a defect or crack. The lower bound property value, or design allowable, is often statistically determined using either A- or B-basis analysis as detailed in the Composite Materials Handbook, CMH-17 [1]. These values represent conservative lower-bound estimates that ensure a specified percentage of the population will meet or exceed the stated property with defined confidence, when taking into account any defects. Material qualification testing considers the variation of test data for a given property under defined conditions so that designers can be confident a material will satisfy the required standards, mitigating the 'weakest link' effect and providing a statistically defensible value.

All measurements have a degree of uncertainty associated with them which effectively represents a range of values within which lies the true value of a given property, with a defined level of confidence. Uncertainty estimation considers the uncertainty in the material property due to the inherent variation of the material, as well as from the equipment and process used in the measurement of materials data, e.g. uncertainty of load cell used to measure load, uncertainty of micrometer used to measure specimen thickness. A- and B-basis design allowables are statistically determined values derived from measured data. Therefore, it is critical that the uncertainty of measurement is considered as this will directly influence the determination of design allowables and performance of any component.

Composite materials, like all materials, possess a degree of variation from a variety of sources which must be considered when designing a component based on test data. A and B-basis analysis use a limited number of specimens to provide a design allowable factoring in the material variation. There are three common sources of variation in a test dataset:

- **Batch variation owing to material composition.** The matrix and fibre reinforcement both contribute differently to the overall properties of a composite material. Any variation in their properties, as they are manufactured, as well as how they interact with each other will create variations between batches.

- **Panel variation due to processing.** There are many aspects of processing that can affect the measured properties, from consistency of laying up along to methods of curing. For instance, when curing composite panels, subtle differences in time, temperature and pressure can lead to observed variation.

- **Specimen variation**. Due to the inherent nature of the construct of composite materials there will be variation in properties between specimens from the same panel. This may be a result of how the specimen has been prepared (such as cutting and the application of end tabs) or due to varying stress distributions as a result of differing void and fibre content.

Additionally, the test setup and environment possess sources of uncertainty which will combine and attribute to potential variation between specimens.

This report looks at how total uncertainty of measurement can be compared to B-basis analysis using example datasets from a small test programme.

2 UNDERSTANDING B-BASIS ANALYSIS

A and B-basis values are statistical measures used in material testing and design. Using a limited number of specimens from a test programme and extrapolating for a global population, it represents a strength or property value which only 10% of the property values from specimens are expected not to exceed with a 95% confidence level. This is decreased to only 1% of specimens failing to exceed with 95% confidence for the A-basis value. These concepts are shown graphically in Figure 1. The A and B-basis values are therefore statistically derived values that are used for critical applications in aerospace and structural engineering ensuring reliability and safety.

B-basis value -- A statistically-based material property; a 95% lower confidence bound on the tenth percentile (First percentile for A-basis) of a specified population of measurements. Also a 95% lower tolerance bound for the upper 90% (99% for A-basis) of a specified population. (CMH-17, 8-4)

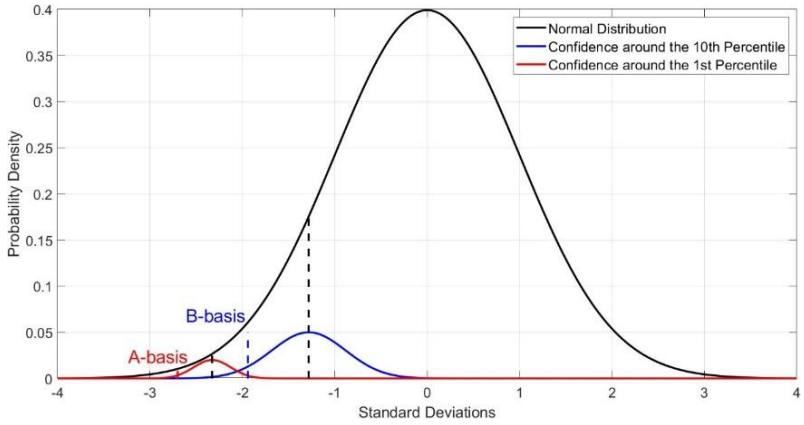


Figure 1 A and B-basis on a normal distribution curve

2.1 SAMPLING REQUIREMENTS FOR B-BASIS ANALYSIS

Although A and B-basis analyses can be undertaken on any number of specimens, guidance provided in the Composite Materials Handbook [1] defines the minimum number of specimens that should be used for the analysis to be valid (Table 1). Figure 2 shows how the specimens could be extracted for a B-basis analysis using a reduced sampling regime.

Table 1 Minimum number of specimens for A and B-basis analysis

Allowable	Sampling	No. of batches	No. of samples
A-basis	Robust	10	75
	Reduced	5	55
B-basis	Robust	10	55
	Reduced	3	18

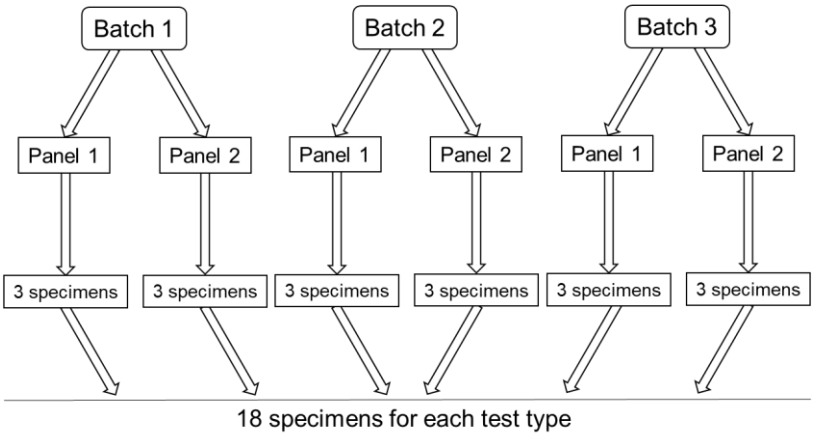


Figure 2 B-basis reduced sampling

2.2 CALCULATING THE B-BASIS VALUE

There are several variations on a basic methodology that can be used for undertaking a B-basis analysis, three are briefly detailed below, as well as when they should be employed, but a more substantive guide is provided in CMH-17 [1]. It is critical to detail which variation has been used as they calculate different B-basis values.

For some datasets it is possible to group them together and analyse the entire grouping as a single dataset. This can only be done for datasets with no distinct cross batch/subset variability, known as unstructured data. Often however, composite materials are structured in that they display anisotropic material properties or have varying properties dependent on which batch the specimens have been drawn from. E.g., Composite materials often exhibit different material properties when tested in different directions (0° vs 90° or warp vs weft) or at different temperatures. Unstructured datasets have been statistically determined to have no significant variation between subsets. The k-sample Anderson-Darling test procedure (CMH-17, Section 8.3.2.2) is used to assess whether two or more groups constituting a population of data are identical and therefore unstructured.

The methods below assume that the dataset is unstructured. Non-parametric methods for A and B-basis analyses are used for structured datasets.

1. The Hanson-Koopmans method: used for sample sizes not exceeding 28 and requires the assumption that the observations are a random sample from a population for which the logarithm of the cumulative distribution function is concave, an assumption satisfied by a large class of probability distributions. Composite strength data largely satisfy this. (CMH 17, 8.3.6.6.4.2)

$$B = x_{(r)}\left[\frac{x_{(1)}}{x_{(r)}}\right]^{k} \qquad (1)$$

$x_{(r)} = the\ r^{th}\ largest\ data\ value, 9\ for\ 18\ specimens$
$x_{(1)} = the\ smallest\ data\ value$
$k = Non-parametric\ B-basis\ factors\ for\ small\ sample\ sizes,$
$1.354\ for\ 18\ specimens$

2. Normal distribution: the most appropriate probability distribution to assign to the data can be assessed either graphically, by producing a histogram of the dataset, or

statistically using a method such as the Kolmogorov-Smirnov or Anderson Darling test. CMH-17 provides guidance on how to use the Anderson-Darling test for this purpose in Section 8.3.6.5.1.2.

$$B = \bar{x} - k_b s \quad (2)$$

$\bar{x} = sample\ mean$
$s = sample\ standard\ deviation$
$k_b = one\ sided\ tolerance\ limit\ factor\ (normal\ distribution), 1.974\ for\ 18\ specimens$

3. Weibull distribution: a Weibull probability distribution can be fitted to the data, if appropriate, by calculating the scale and shape parameters. (Section 8.3.6.5.2.1)

$$B = \hat{\alpha}(0.10536)^{1/\hat{\beta}}\left(exp\left\{\frac{-V}{\hat{\beta}\sqrt{n}}\right\}\right) \quad (3)$$

$\hat{\alpha} = scale\ parameter, maximum\ likelihood\ estimation$
$\hat{\beta} = shape\ parameter, maximum\ likelihood\ estimation$
$V_b = one\ sided\ tolerance\ limit\ factor\ (Weibull\ distribution), 5.605\ for\ 18\ specimens$
$n = number\ of\ specimens$

3 DETERMINATION OF MEASUREMENT UNCERTAINTY

To have confidence in any measurement, the uncertainty of that measurement should be quantified. Uncertainty can be defined as 'a *parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand'* [2]. It quantifies, to a certain confidence level, that the true value of a measurement lies between an upper and lower bound. Composite materials exhibit variation between specimens and individual specimens cannot be retested. Therefore, with respect to composite material testing, the uncertainty is a range of values that the measurand of subsequent tests with an identical test setup will fall between to a defined confidence level.

An uncertainty budget can be used which combines individual sources of uncertainty from the test setup and procedure, and provides a total uncertainty of the measurement. Figures 3 & 4 provide general guidance on how to quantify the uncertainty contribution following guidance found in GUM [2] and UNCERT manuals [3].

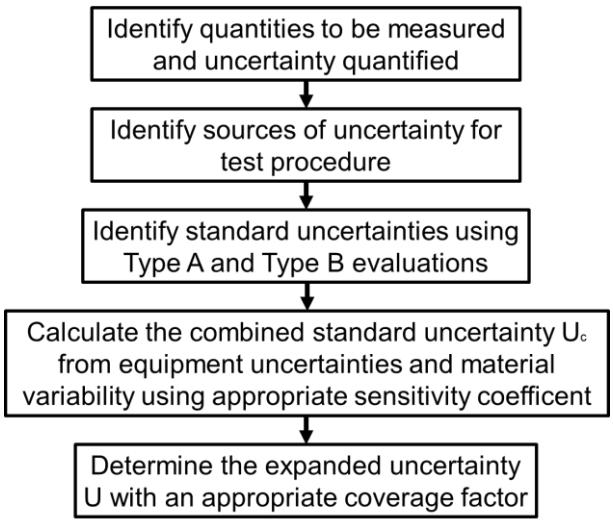


Figure 3 Steps for estimating uncertainty of a measurement.

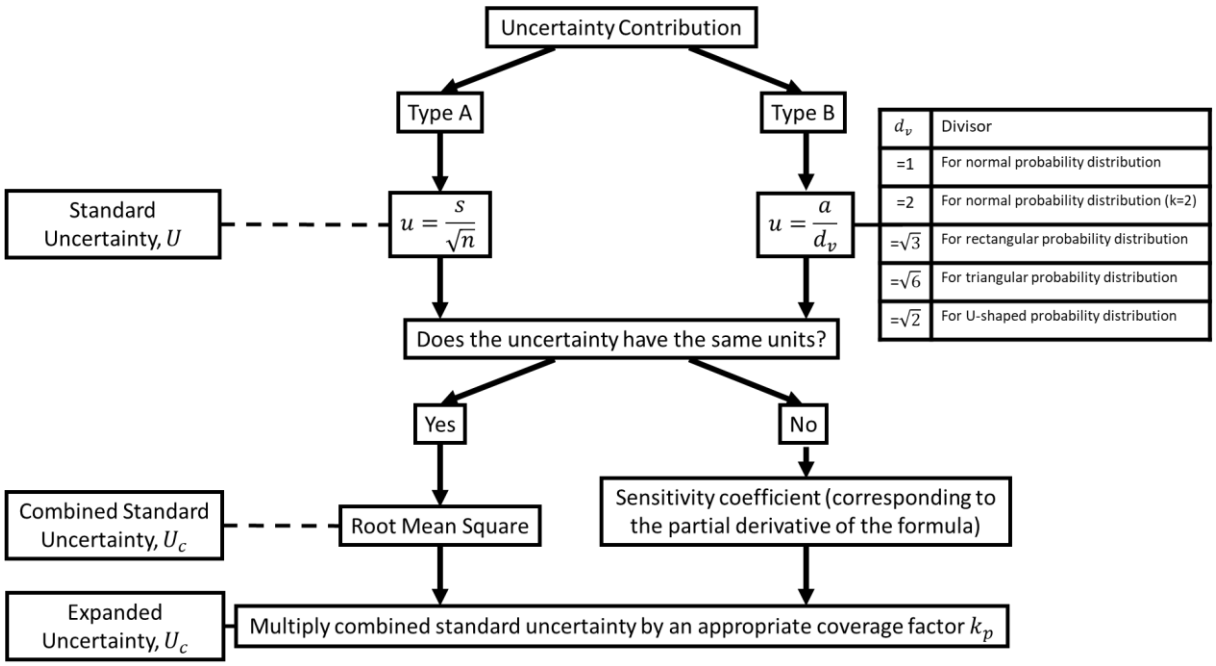


Figure 4 Steps for estimating uncertainty of a measurement.

To support the implementation of the uncertainty budget in a mechanical testing environment, four main sources of uncertainty have been identified and detailed in Table 2. Each of these sources have been examined and, where possible, quantified in intra-laboratory round-robin studies or though experimental analysis.

Table 2 Minimum number of specimens for A and B-basis analysis

Uncertainty Source	**Description**	**Measurand affected**
Material variability	The variability of specimens within a batch or that have been extracted from a panel. Also, takes into account how the data have been analysed	Reported material properties
Dimensioning	Measurements of specimen dimensions	Length, width thickness, radius/diameter etc
Load	Uncertainty associated with the equipment used to carry out the load measurements	Load
Strain measurement	The strain measurement technique used (e.g. strain gauges) to carry out measurements on the specimens	Strain, ε

Sources of uncertainty can then be identified to support the construction of an uncertainty budget. Some of the sources of uncertainty are shown in Figure 5 but this list is not exhaustive. Other sources of uncertainty can be eliminated or reduced through good practice.

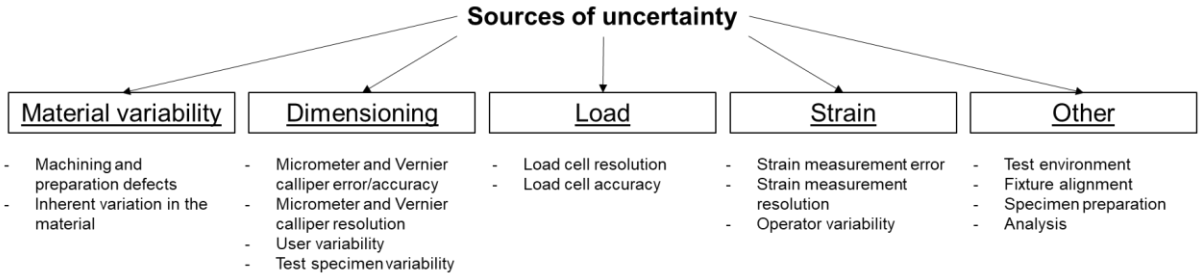


Figure 5 Common sources of uncertainty in mechanical testing.

Once the uncertainty contributions for each source have been quantified it is possible to combine them to provide a total uncertainty of the measurement.

4 DATA FOR ANALYSIS

4.1 SELECTION OF MATERIAL AND TEST METHODOLOGY

Material was identified and supplied by Hexcel to carry out reduced sampling for B-basis analysis. The panels, from three batches, were processed in-house in separate oven cure cycles. A quasi-isotropic layup was selected which allowed for each panel to be used for various in-plane test methods. Tension (ISO 527-4 [4]), compression (ISO 14126 [5]) and open hole tension (ASTM D5766 [6]) datasets were produced for each panel. Axial strain was measured using a combination of strain gauges and clip-on extensometry for the tensile tests and strain gauges for the compression tests. The full datasets, including stress-strain plots of 18 specimens per test method, are shown in Appendix A, Figure A1.

4.2 REPRESENTATION OF DATA

To visualize the probability distributions for each dataset histograms representing the data are shown in Figures 6-8. The total area of all the bins adds up to the total number of test specimens in the dataset (i.e. 18). The probability density is given by Equation 4.

$$Probability\ Density\ V_i = \frac{C_i}{NW_i} \tag{4}$$

$C_i = number\ of\ elements\ in\ the\ bin$
$W_i = width\ of\ the\ bin$
$V_i = bin\ value$
$N = number\ of\ specimens$

A probability distribution curve has been fitted to each dataset with the B-basis value, calculated using the associated method, indicated on the plot. Additionally, the total uncertainty of the dataset is indicated as well as the uncertainty contribution from the test methodology and procedure around the mean value of the dataset.

The reported total uncertainty is based on a standard uncertainty multiplied by a coverage factor k = 2.11, providing a level of confidence of 95 %.

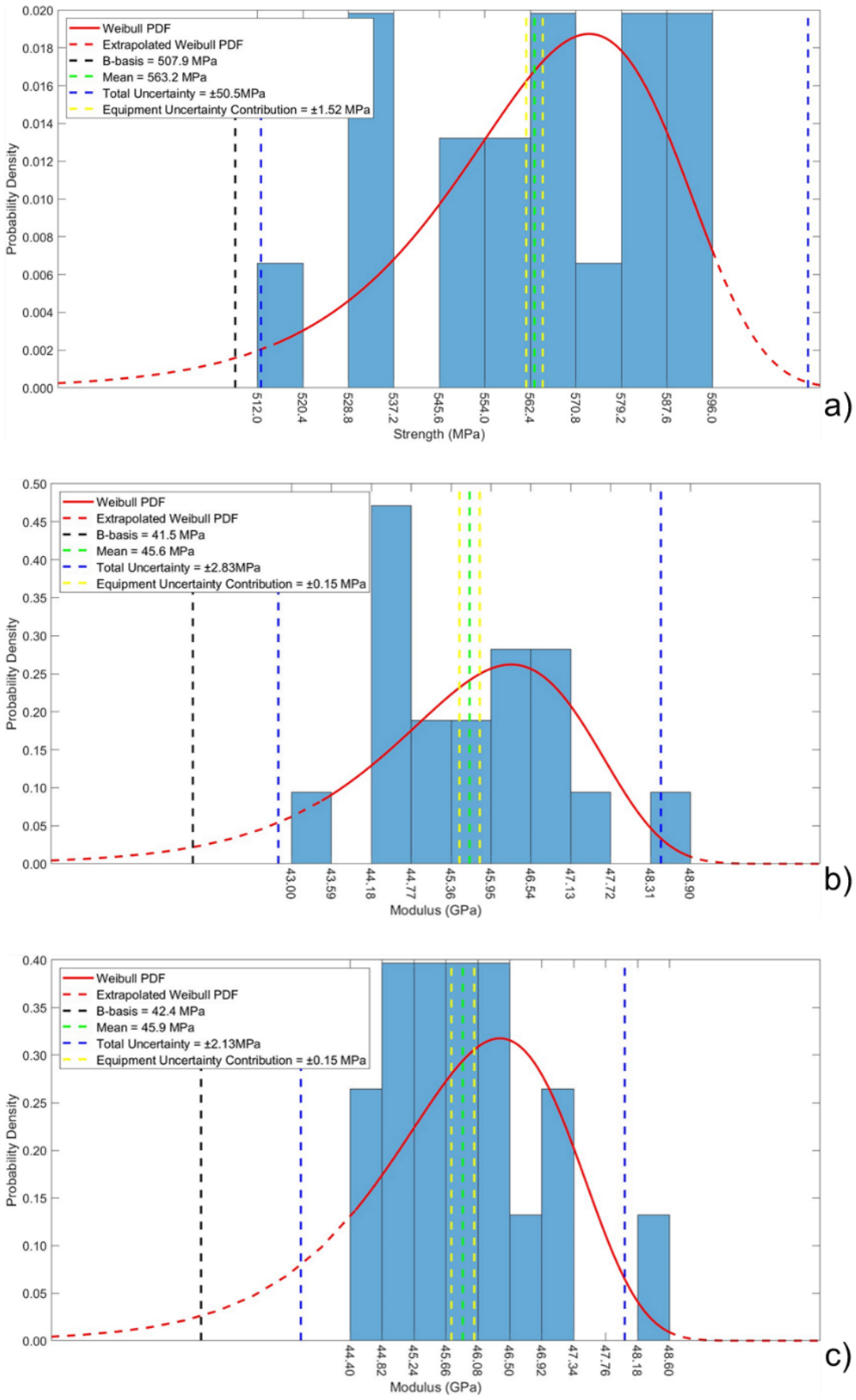


Figure 6 Histogram plots with Weibull distribution curve for tension dataset, a) Strength, b) Modulus (strain gauge), Modulus (Extensometer)

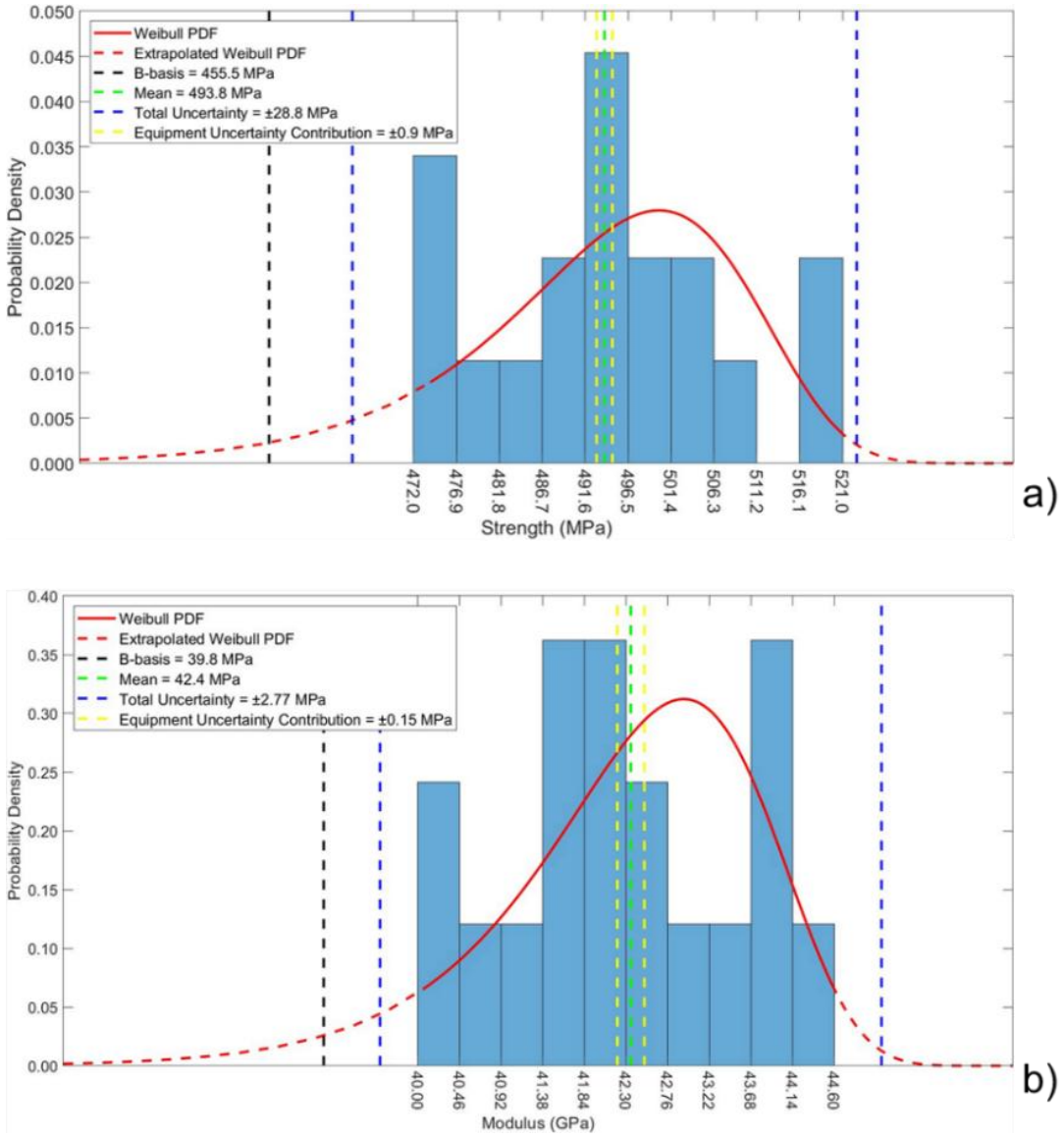


Figure 7 Histogram plots with Weibull distribution curve for compression dataset, a) Strength, b) Modulus (strain gauge)

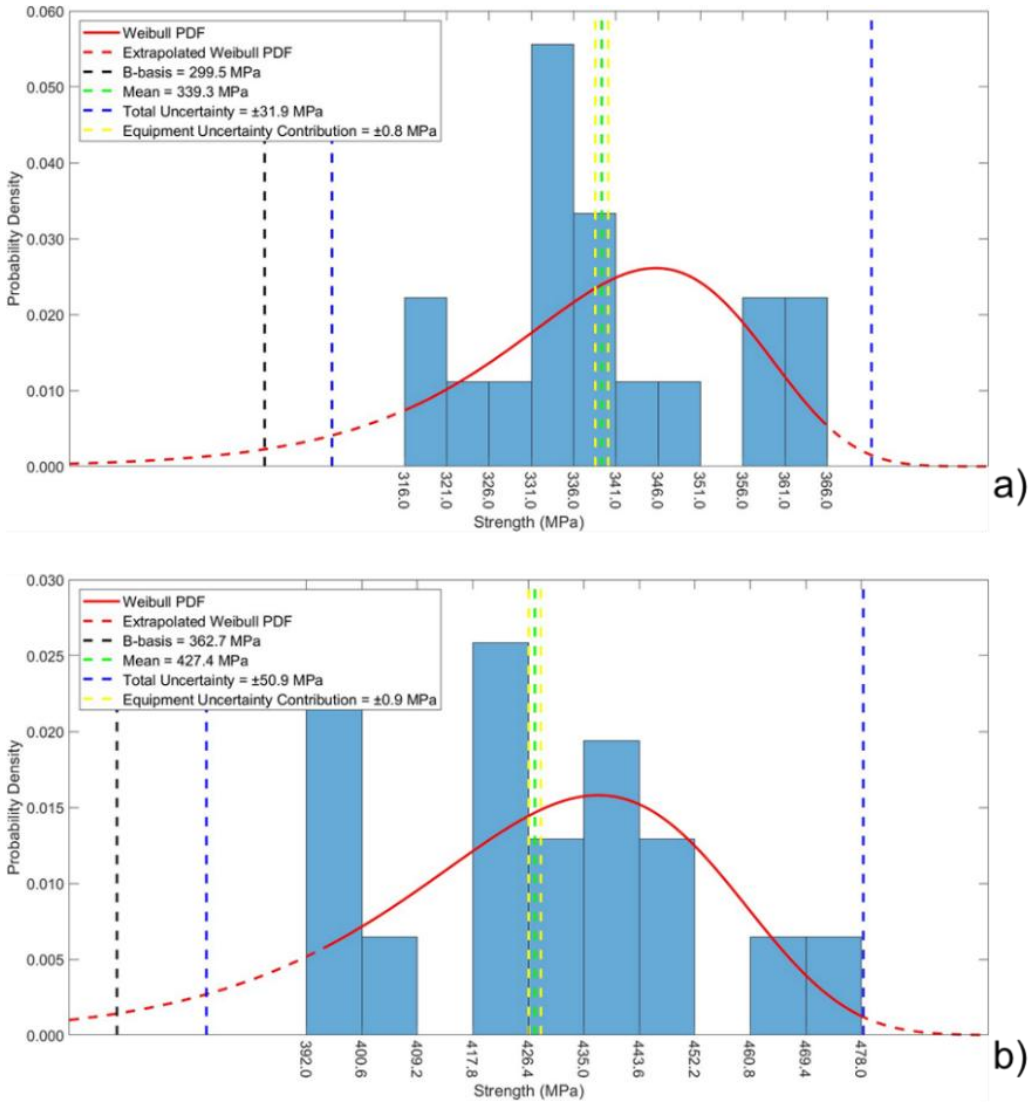


Figure 8 Histogram plots with Weibull distribution curve for open hole tension dataset, a) Initial failure strength, b) Ultimate strength

The histogram plots show that a Weibull probability distribution is most appropriate for the datasets. B-basis values for all datasets have been calculated following the methods described in Section 1.3 for comparison.

Table 3 Statistical values for all datasets showing the calculated B-basis values and a comparison to total uncertainty.

Test Type	Tension			Compression		Open Hole Tension	
Property	**Strength**	**Modulus (Strain Gauges)**	**Modulus (Extens-ometer)**	**Strength**	**Modulus**	**Initial Failure Strength**	**Ultimate Strength**
	MPa	**GPa**	**GPa**	**MPa**	**GPa**	**MPa**	**MPa**
Mean	563.1	45.63	45.88	493.8	42.35	339.3	427.4
St. Dev	23.9	1.34	1.01	13.6	1.31	14.5	23.6
CV (%)	4.2	2.9	2.2	2.8	3.1	4.3	5.5
Lower Uncertainty Bound	512.6	42.80	43.75	465.0	39.58	307.4	376.5
Equipment Uncertainty (% of total uncertainty)	2.8	5.3	6.9	3.1	5.4	2.5	1.9
B-Basis Value (% difference compared to lower uncertainty bound)							
Hanson-Koopmans	499.2 (-2.6)	42.78 (-0.1)	44.05 (0.7)	467.4 (0.5)	39.38 (-0.5)	309.9 (0.8)	385.2 (2.3)
Normal Distribution	515.9 (0.6)	42.99 (0.4)	43.89 (0.3)	466.9 (0.4)	39.77 (0.5)	310.6 (1.0)	380.9 (1.2)
Weibull Distribution	507.9 (-0.9)	41.54 (-3.0)	42.44 (-3.0)	455.5 (-2.0)	38.96 (-1.6)	299.5 (-2.6)	362.6 (-3.7)

As Table 3 shows, there is close agreement between total uncertainty and all calculated B-basis values. This evidence supports the robustness of both B-basis analysis as well as determination of uncertainty of the measurement further validating both methods as a tool for design and modelling of composite material components.

Table 4 Comparison of B-basis values against lower uncertainty bound.

B-basis analysis method	Difference compared to lower uncertainty bound (%)
All	-0.53
Hanson-Koopmans	0.16
Normal Distribution	0.64
Weibull Distribution	-2.40

5 CONCLUSION

A and B-basis allowables are important quantities for the design of critical infrastructure using composite materials allowing test data to be effectively used for design allowables. The similarity in values for uncertainty and B-basis analyses support the robustness of statistically determined design values as well as validating uncertainty determination as an additional tool assuming the sources of uncertainty have been adequately quantified.

REFERENCES

[1] Composite Materials Handbook – Volume 1, CMH-17
[2] Evaluation of measurement data – Guide to the expression of uncertainty, JCGM 100:2008,
[3] Manual of Codes of Practice for the Determination of Uncertainties in Mechanical Tests on Metallic Materials, 2000
[4] ISO 527-4, Plastics — Determination of tensile properties, Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites
[5] ISO 14126, Fibre-reinforced plastic composites — Determination of compressive properties in the in-plane direction
[6] ASTM D5766, Open-Hole Tensile Strength of Polymer Matrix Composite Laminates

APPENDIX A

TEST MATERIALS

The test materials were supplied by Hexcel as 600 mm x 600 mm HexPly® M79-LT 35%/UD600+PES/CHS-50K unidirectional prepreg laminate sheets in three batches as shown in Table A1. The laminate sheets were cut in half prior to curing to enable two panels to be processed from each batch in a quasi-isotropic $(+45°, -45°, 0°, 90°)_s$ layup. Each panel was cured individually for 6 hours at 80 °C under vacuum.

NPL Materials Index Register (MIR) codes were allocated to each panel in accordance with NPL quality procedures. These codes are used to identify individual test specimens.

Table A1 Details of test panels

Hexcel Batch ID	NPL MIR Code
30170059509	1AKHZ
	3AKHZ
30170059409	1AKIA
	2AKIA
30170059407	1AKIB
	2AKIB

After each cure cycle, the following quality assessment tests were carried out on each panel to identify any panels that should be quarantined and removed from the test programme:

- Visual inspection of the panel to identify surface defects.
- Ultrasonic c-scan to identify large internal manufacturing defects.
- Dynamic mechanical analysis (DMA) to determine the glass transition temperature, T_g.
- Microwave-assisted acid digestion to determine fibre, resin and void content.

TEST EQUIPMENT USED AND CALIBRATION INFORMATION

A summary of the test equipment used, and the calibration details is given in Table A2. All measuring equipment used had certified accredited calibrations or were verified at the time of testing using an accredited calibration device.

Table A2 Summary of test equipment and calibration details

Item	Equipment	Accredited calibration certificate no.	Calibration date	Standard uncertainty
1	Instron 5985, no. B14900 100 kN load cell, no. 134916	E323092723115633	Sep. 2023	± 0.19 %
2	Mitutoyo digital micrometer, no. 11010976	CN338156	Jun. 2023	± 0.002 mm
3	Mitutoyo digital Vernier caliper, no. B18178745	CN341067	Sep. 2023	± 0.002 mm
4	National Instruments cDAQ 9178/1720837	Verified using items 5 & 6	N/A	N/A
5	Meatest precision resistance decade box M632/625051	CN336411	Apr. 2023	± 0.001 % + 1 LSD

Table A2 continued Summary of test equipment and calibration details.

Item	Equipment	Accredited calibration certificate no.	Calibration date	Standard uncertainty
6	Fluke 726 precision calibrator, no. 2877125	CN336476	Apr. 2023	-
7	Epsilon Biaxial Extensometer, no. 3560-BIA-050M-005-HT1	Verified using item 8 to ISO 9513, Class 0.5	N/A	0.3 %
8	Laser interferometer calibration rig	2020020411-1	Feb. 2020	± 0.1 µm
9	Rotronic A2 Hygromer, no. 10048 003	2023040098	Jun. 2023	± 0.1 °C

*coverage factor *k*=2, providing a level of confidence of approximately 95%

TEST PROCEDURE

All mechanical tests were carried out in a laboratory atmosphere controlled at 23 ± 2 ºC and 50 ± 10 % relative humidity (RH). The conditions were monitored at the start of every test using a calibrated hygrometer to ensure they remained within these ranges.

Tension

Tensile tests were undertaken in general accordance with the method described in ISO 527-4, on an Instron 5985 mechanical test machine fitted with a 100 kN load cell. Specimens were machined to 250 mm x 25 mm, and 2 mm thick 50 mm long Tufnol® 10G/40 end-tabs were bonded to the material using 3M ScotchWeld® EC-9323 adhesive. The specimens were gripped using a pair of hydraulic wedge-action grips fitted with serrated grip faces with a nominal gripping pressure of 50 bar. The specimens were loaded at a crosshead speed of 2 mm per minute until failure.

Axial and transverse strain was measured using biaxial 5 mm strain gauges on opposite faces of the specimens and averaged. A biaxial extensometer was used to measure axial strain and was removed at 0.4 % axial strain to prevent damage to the extensometer at failure. The strain gauges remained attached until failure.

The modulus values were calculated over the strain range 0.05-0.25 %, from the average strains measured on opposite faces. The tensile modulus was calculated using the slope method which uses a least squares regression analysis to determine the modulus across all values within the 0.05-0.25 % strain range. Tensile strength was calculated using the maximum load divided by the cross-sectional area.

Compression

Compression tests were undertaken in general accordance with the method described in ISO 14126, on an Instron 5985 mechanical test machine fitted with a 100 kN load cell. Specimens were machined to 125 mm x 25 mm, and 2 mm thick 50 mm long Tufnol® 10G/40 end-tabs were bonded to the material using 3M ScotchWeld® EC-9323 adhesive. The specimens were ground flat at the end faces to ensure even loading. The specimens were loaded using a four-pillar die-set. The specimens were loaded at a crosshead speed of 1 mm per minute until failure.

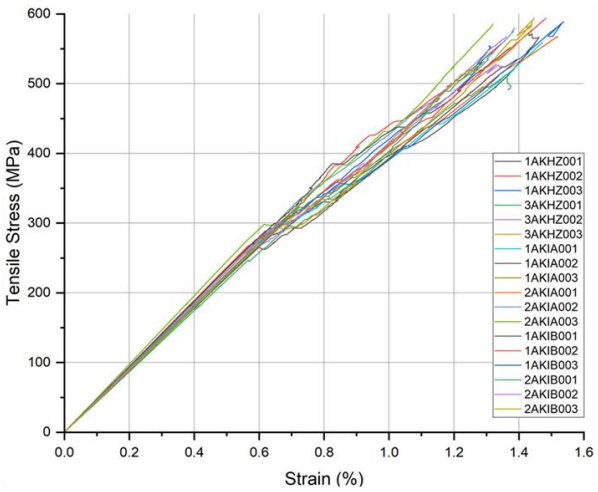
Axial and transverse strain was measured using biaxial 5 mm strain gauges on opposite faces of the specimens and averaged. The strain gauges remained attached until failure.
The modulus values were calculated over the strain range of 0.05-0.25 %, from the average strains measured on opposite faces. The compressive modulus was calculated using the slope method which uses a least squares regression analysis to determine the modulus across all values within the 0.05-0.25 % strain range. Compressive strength was calculated using the maximum load divided by the cross-sectional area.

Open Hole Tension

Open Hole Tension tests were undertaken in general accordance with the method described in ASTM D5766, on an Instron 5985 mechanical test machine fitted with a 100 kN load cell. Specimens were machined to 250 mm x 36 mm. The specimens were ground flat at the end faces to ensure even loading. A 6 mm hole was machined at the centre of each specimen and visually inspected prior to testing. The specimens were loaded at a crosshead speed of 2 mm per minute until failure.

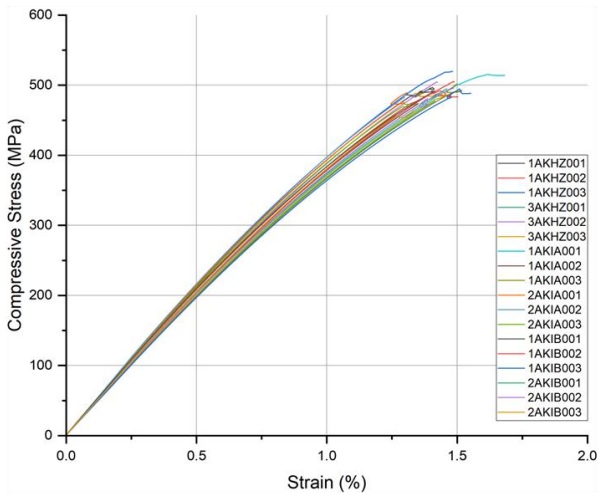
Tensile strength was calculated using the maximum load divided by the cross-sectional area.

a)



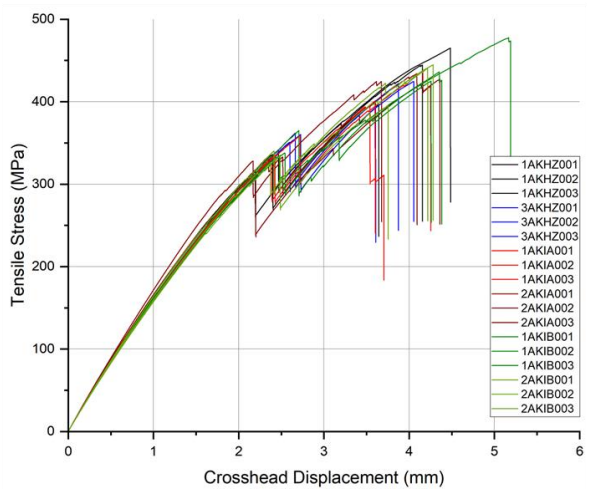
Specimen ID	Strength (MPa)	Modulus SG (GPa)	Modulus Ext (GPa)
1AKHZ001	574.2	46.03	45.92
1AKHZ002	563.1	47.24	46.82
1AKHZ003	556.3	46.88	47.00
1AKIA001	560.5	45.38	45.72
1AKIA002	536.1	44.23	44.82
1AKIA003	533.2	44.20	44.85
1AKIB001	553.1	46.79	45.67
1AKIB002	595.3	45.27	45.14
1AKIB003	590.3	44.58	45.17
2AKIA001	569.4	46.83	47.07
2AKIA002	581.5	45.97	46.16
2AKIA003	587.0	48.82	46.22
2AKIB001	547.1	45.31	45.46
2AKIB002	569.0	46.06	46.19
2AKIB003	580.1	44.47	48.58
3AKHZ001	515.1	43.45	45.31
3AKHZ002	529.3	45.41	44.47
3AKHZ003	596.0	44.49	45.29

b)



Specimen ID	Strength (MPa)	Modulus (GPa)
1AKHZ001	497.6	43.93
1AKHZ002	492.6	42.72
1AKHZ003	520.5	44.00
1AKIA001	516.5	41.84
1AKIA002	474.5	42.05
1AKIA003	502.8	40.40
1AKIB001	492.7	43.18
1AKIB002	506.1	41.66
1AKIB003	495.6	40.08
2AKIA001	490.9	44.01
2AKIA002	484.0	44.58
2AKIA003	474.6	43.44
2AKIB001	494.4	41.91
2AKIB002	496.9	41.76
2AKIB003	473.9	42.15
3AKHZ001	478.5	40.75
3AKHZ002	506.9	42.62
3AKHZ003	489.5	41.26

c)



Specimen ID	Initial Failure Strength (MPa)	Ultimate Strength (MPa)
1AKHZ001	341.2	465.2
1AKHZ002	336.2	396.6
1AKHZ003	317.4	444.6
3AKHZ001	361.9	422.0
3AKHZ002	350.6	400.2
3AKHZ003	360.3	424.5
1AKIA001	332.6	401.3
1AKIA002	335.5	395.3
1AKIA004	358.3	437.4
2AKIA001	316.4	427.3
2AKIA002	333.8	434.1
2AKIA003	328.1	424.8
1AKIB001	333.1	477.7
1AKIB002	337.8	425.1
1AKIB003	365.0	436.3
2AKIB001	325.5	444.8
2AKIB002	333.9	395.3
2AKIB003	340.3	440.9

Figure A1 a) Tension, b) Compression, c) Open Hole Tension datasets.