Statistical based approach to material identification of composite materials

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Abstract: Experimentally investigated material properties of unidirectional carbon fibre composite samples manufactured in an autoclave were subjected to the statistical analysis. The Maximum normed residual method was used for the identification of outliers within each batch. Furthermore, the k-sample Andreson-Darling test was used to determine whether the properties from different batches are combinable. Combinable material properties were again tested for outliers. Resulting set of data was tested using the Anderson-Darling goodness-of-fit test in order to evaluate whether the approximation by the Normal distribution, the Lognormal distribution or the Weibull 2-parameter distribution would be appropriate. Material properties were compared within one batch as well as between batches.

Keywords: Carbon fibre composite; Anderson-Darling; distribution; identification; statistic.

1 Introduction

Due to the benefits in form of high stiffness and strength compared to low density, composite materials are widely used in many applications in sport, automotive, marine, and aerospace industries. A composite production process consists of the positioning of dry or pre-impregnated fibres, a potential resin infusion and a curing process. The positioning of fibres usually performed by human workers or robots introduces certain material variability factor into each composite part which may result in stiffness and strength variability of a final structure.

This material variability may result from number of sources as batch-to-batch variability of raw materials, batch-to-batch variability caused by operators, batch intrinsic variability and testing variability [1]. For the approximation of material property variability, the Normal distribution, the Lognormal distribution and the 2-parameter Weibull distribution are the most appropriate selections [1]. The Normal distribution was used in [2] for the approximation of stiffness and strength parameters of carbon fibre composite. Results of the approximation were later implemented into the finite element method software and used for a fatigue simulation. The Weibull distribution was used in [3], [4], and [5]. The paper [3] was focused on the approximation of strength of unidirectional carbon fibre samples and on the parameter estimation of Weibull distribution using the linear regression. The Maximum likelihood method was used for the Weibull distribution parameters estimation in [4]. The distribution was used for the approximation of bending stiffness of carbon fibre composite having layup $[0/90]_{2S}$. Also in [5], the Weibull distribution with the Maximum likelihood method was used for the approximation of bamboo fibre strength.

For the purposes of using own manufactured composite material for research of riveted joints, this work was focused on manufacturing of test samples and the investigation of their material properties. Furthermore, the detection of outliers, the test for combinability of results obtained from measurements from different batches, and the selection of appropriate statistical distribution was carried out.

2 Preparation of test samples

Two unidirectional (UD) carbon fibre plates were manufactured by author in the ASC Econoclave autoclave (see Fig. 1) from non-crimped fabric (NCF) Saertex with fibres TENAX-J IMS60 E13 24K and from epoxy resin MSG L285. For the manufacturing process, Airbus patented Vacuum Assisted Process (VAP®) was selected [6]. This process is characterized by using VAP® membrane which brings a benefit of better resin distribution during the infusion resulting in better fibre to resin volume ratio than in case of a standard infusion procedure. Furthermore, this process reduces the amount of air bubbles closed in composite and therefore decreases the material property variability.

Test samples were cut using a CNC milling machine from different plates. For the testing, dimensions following ASTM D3039 were determined as 200 mm × 25 mm × 2.6 mm. Placement and orientation of samples on the cut plates were selected in order to obtain samples having fibre orientation 0°, 45° and 90° with respect to the longer edge of a sample (see Fig. 2). Six samples per each fibre orientation and batch were created and numbered to guarantee full traceability.

Dimensions and weight of each sample were measured for the purposes of the fibre volume ratio calculation and the sample stiffness evaluation. Resulting mean fibre volume ratios with standard deviations for each sample batch are listed in Tab. 1.

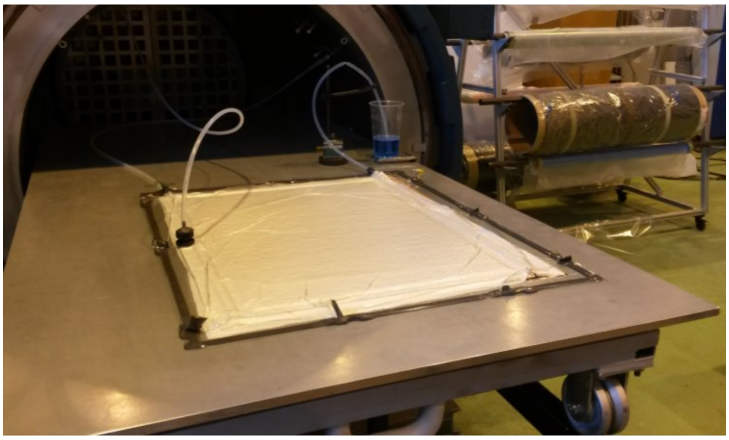

Fig. 1: VAP® manufacturing process of UD carbon fibre plate.

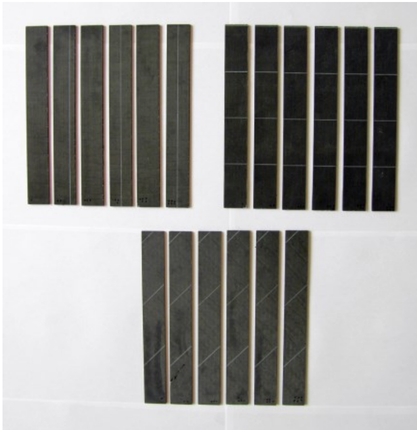

Fig. 2: Example of sample batch having fibre orientation 0°, 90° and 45°.

Tab. 1: Mean fibre volume ratios and standard deviations of fibre volume ratios of test samples.

Samples	Mean fibre volume ratio [%]	Standard deviation of fibre volume ratio [%]
0° fibre orientation - Batch 1	60.7	1.5
90° fibre orientation - Batch 1	60.9	0.4
45° fibre orientation - Batch 1	60.6	0.3
0° fibre orientation - Batch 2	63.7	1.2
90° fibre orientation - Batch 2	63.7	0.2
45° fibre orientation - Batch 2	64.7	0.2

3 Theoretical background

3.1 Maximum normed residual test

The Maximum normed residual test (MNR) is a test for the identification of outliers in a set of unstructured data $x_1, x_2, \ldots, x_n$. The MNR can be calculated as:

$$\mathrm{MNR} = \max_i \frac{|x_i - \bar{x}|}{s}, \qquad i=1, 2, \ldots, n \tag{1}$$

where $\bar{x}$ denotes to the sample mean and s to the standard deviation. This number is compared to the critical value (CV) calculated for the sample size n as:

$$\mathrm{CV} = \frac{n-1}{\sqrt{n}} \sqrt{\frac{t^2}{n-2+t^2}}, \tag{2}$$

where

$$t = [1 - \alpha/2n]. \tag{3}$$

α is the significance level and recommended $\alpha = 0.05$ for this test. If the MNR is lower than CV then it can be concluded that no outlier was detected. Otherwise, the sample associated with highest $|x_i - \bar{x}|$ is detected as an outlier. Once the outlier is detected, it has to be removed from calculations and the MNR test must be repeated [1].

3.2 The k-sample Anderson-Darling test

The k-sample Anderson-Darling test is used for the verification of the hypothesis whether the samples from different batches can be combined into one group of observations $z_1, z_2, \ldots, z_L$. This hypothesis is confirmed if the ADK number is lower than critical value ADC. ADK can be calculated as [1]:

$$\mathrm{ADK} = \frac{n-1}{n^2(k-1)} \sum_{i=1}^{k} \left\{ \frac{1}{n_i} \sum_{j=1}^{L} h_j \frac{\left(nH_{ij} - n_i H_j\right)^2}{H_j(n-H_j) - nh_j/4} \right\}, \tag{4}$$

where

n	is the number of combined samples,
k	is the number of batches,
L	is the number of observations,
h_j	is the number of values in the combined samples equal to z_j,
H_j	is the number of values in the combined samples less than z_j plus one half the number of values in the combined samples equal to z_j,
F_{ij}	is the number of values in the i-th group which are less than z_j plus one half the number of values in this group which are equal to z_j.

The critical value ADC can be evaluated as:

$$\mathrm{ADC} = 1 + \sigma_n \left[1.645 + \frac{0.678}{\sqrt{k-1}} - \frac{0.362}{k-1}\right], \tag{5}$$

where

$$\sigma_n^2 = \mathrm{Var}(\mathrm{ADK}) = \frac{an^3 + bn^2 + cn + d}{(n-1)(n-2)(n-3)(k-1)^2}, \tag{6}$$

$$
\begin{aligned}
a &= (4g-6)(k-1) + (10-6g)S, \\
b &= (2g-4)k^2 + 8Tk + (2g-14T-4)S - 8T + 4g - 6, \\
c &= (6T+2g-2)k^2 + (4T-4g+6)k + (2T-6)S + 4T, \\
d &= (2T+6)k^2 - 4Tk,
\end{aligned} \tag{7}
$$

$$S = \sum_{i=1}^{k} \frac{1}{n_i}, \tag{8}$$

$$T = \sum_{i=1}^{n-1} \frac{1}{i}, \tag{9}$$

3.3 Anderson-Darling goodness-of-fit test for Normal and Lognormal distribution

For the hypothesis whether the observations can be approximated by the Normal or the Lognormal distribution, the Anderson-Darling goodness-of-fit test can be used. The hypothesis is confirmed with five percent of risk of an error if the Observation significance level (OSL) is lower than 0.05. The OSL can be calculated for values [1]:

$$z_i = \frac{x_i - \bar{x}}{s}, \quad \text{in case of the Normal distribution,} \tag{10}$$

$$z_i = \frac{\ln(x_i) - \bar{x}_L}{s_L}, \quad \text{in case of the Lognormal distribution,} \tag{11}$$

where $\bar{x}$ and s are the sample mean and the standard deviation, respectively. $\bar{x}_L$ and s_L denote to the mean and the standard deviation calculated from observations $\ln(x_i)$, respectively.

The OSL is then evaluated as follows:

$$\mathrm{AD} = \sum_{i=1}^{n} \left[\frac{1-2i}{n}\{ln[F_0(z_i)] + \ln[1 - F_0(z_{n+1-i})]\}\right] - n, \tag{12}$$

$$\mathrm{AD}^* = \left[1 + \frac{4}{n} - \frac{25}{n^2}\right]\mathrm{AD}, \tag{13}$$

$$\mathrm{OSL} = 1/\{1 + \exp[-0.48 + 0.78\ln(\mathrm{AD}^*) + 4.58\mathrm{AD}^*]\}. \tag{14}$$

3.4 Anderson-Darling goodness-of-fit test for 2-parameter Weibull distribution

For the hypothesis whether the observations can be approximated by the 2-parameter Weibull distribution, the Anderson-Darling goodness-of-fit test can be used as well. The OSL can be calculated for values [1]:

$$z_i = \left(\frac{x_i}{\hat{\alpha}}\right)^{\hat{\beta}}, \tag{15}$$

where $\hat{\alpha}$ and $\hat{\beta}$ represent the scale and the shape parameters of the Weibull distribution, respectively [7]. The OSL is then evaluated as follows:

$$\mathrm{AD} = \sum_{i=1}^{n} \left[\frac{1-2i}{n}\{ln[1 - \exp(-z_i)] - z_{n+1-i}\}\right] - n, \tag{16}$$

$$\mathrm{AD}^* = \left[1 + 0.2/\sqrt{n}\right]\mathrm{AD}, \tag{17}$$

$$\mathrm{OSL} = 1/\{1 + \exp[-0.10 + 1.24\ln(\mathrm{AD}^*) + 4.48\mathrm{AD}^*]\}. \tag{18}$$

4 Experiments and evaluation

Experiments were carried out in the testing machine Zwick/Roell Z050. All test samples were subjected to tensile loading with standard head speed rate of 2 mm/min. Except of samples having 0° fibre orientation (the ultimate load of these samples was beyond the capacity of the testing machine), samples were loaded up to the failure. The force was measured using load cell and the displacement using an extensometer. The clamp length was 100 mm and the extensometer measurement area was 60 mm long. In total, 36 experiments with samples having 3 different fibre orientations were performed (see fig. 3).

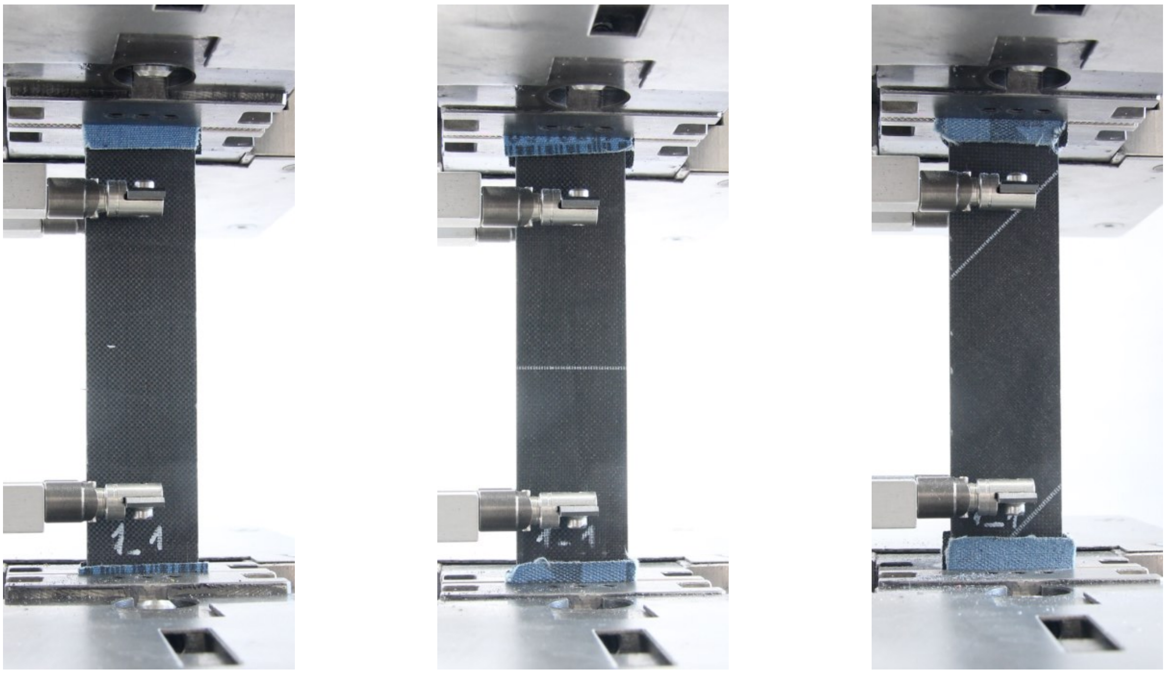

Fig. 3: Experiments with samples having 0° (left), 90° (middle) and 45° (right) fibre orientations.

Investigated force-displacement curves were transformed into the nominal stress-strain curves. In fig. 4, the stress-strain curves for samples having 0° fibre orientation are displayed. Samples were loaded up to 20 kN (approx. 320 MPa). Fig. 5 displays samples having 90° fibre orientation and fig. 6 displays samples having 45° fibre orientation. Here, the non-linear material behaviour of the composite during the shear loading can be observed.

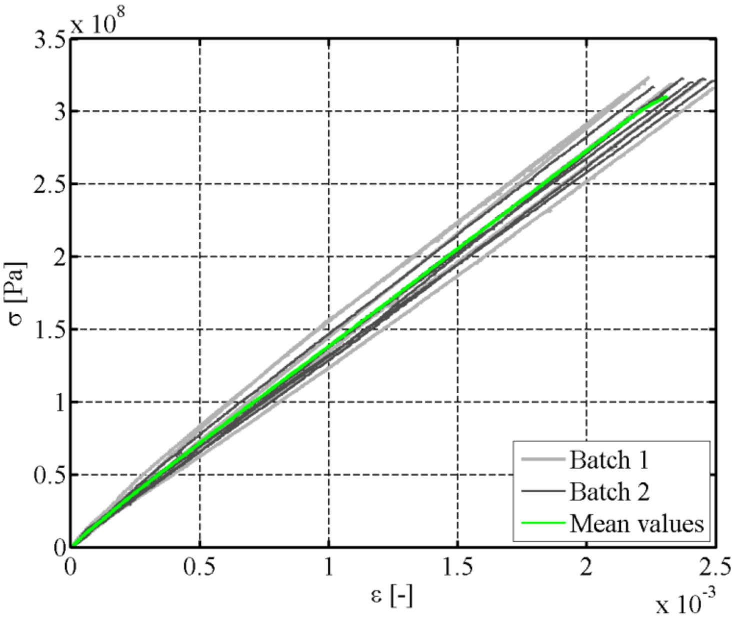

Fig. 4: Stress-strain curves for samples having 0° fibre orientation and their mean values.

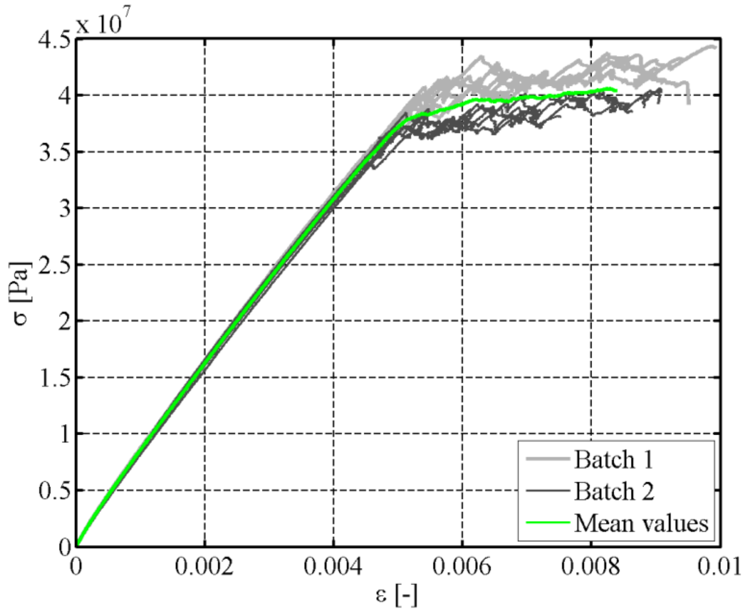

Fig. 5: Stress-strain curves for samples having 90° fibre orientation and their mean values.

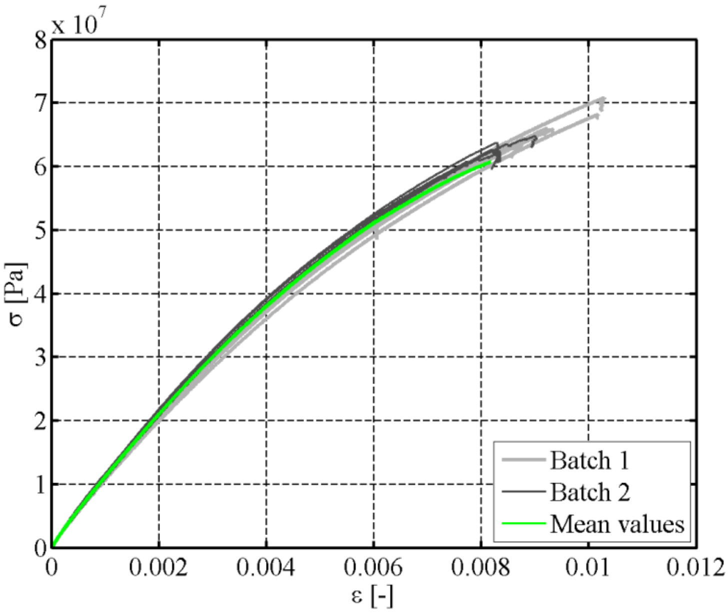

Fig. 6: Stress-strain curves for samples having 45° fibre orientation and their mean values.

From the stress-strain curves, stiffness and strength (except of samples having 0° fibre orientation) parameters of the composite material were determined. All values were tested by the MNR test for the detection of outliers. Only the strength of the sample no. 2 having 45° fibre orientation from the batch 1 was detected as an outlier. Therefore, it was not included into further calculations. The summary of the mean values and the standard deviations calculated from the rest of samples for each batch and fibre orientation are listed in Tab. 2.

Tab. 2: Mean values and standard deviations of stiffness and strength parameters.

Samples	Stiffness mean [MPa]	Stiffness standard deviation [MPa]	Strength mean [MPa]	Strength standard deviation [MPa]
0° fibre orientation - Batch 1	134724.3	5409.1	-	-
0° fibre orientation - Batch 2	132339.4	4562.4	-	-
90° fibre orientation - Batch 1	7671.6	96.8	43.1	1.0
90° fibre orientation - Batch 2	7568.9	118.6	39.5	0.9
45° fibre orientation - Batch 1	12612.6	198.0	67.0	2.5
45° fibre orientation - Batch 2	12916.8	279.4	62.5	1.9

Furthermore, the k-sample Anderson-Darling test was applied in order to determine whether the data obtained from measurements on the sample batches 1 and 2 can be combined and assumed as one set of data. Calculated ADK and ADC numbers are listed in Tab. 3. Due to the requirement to fulfil the ADK < ADC condition, it can be concluded that the stiffness parameters can be combined into one set of data and the strength cannot be combined into one set of data. Therefore, only the stiffness parameters were possible to subject to further tests because the strength parameters would have to be tested separately, which would lead to unprecise results due to the low number of tests within batches.

Tab. 3: Results of k-sample Anderson-Darling test.

Samples	ADK for stiffness param. [-]	ADC for stiffness param. [-]	ADK for strength param. [-]	ADC for strength param. [-]
0° fibre orientation	0.5109	2.2882	-	-
90° fibre orientation	1.4161	2.2882	4.7827	2.2882
45° fibre orientation	2.2154	2.2882	3.7754	2.2710

Corresponding stiffness parameters from the batches 1 and 2 were combined into sets of data and again tested for outliers using the MNR test. No outliers were detected in any of the data sets. Therefore, data were tested by the Anderson-Darling goodness-of-fit tests in order to evaluate the hypothesis whether they can be approximated by the Normal, the Lognormal or the 2-parameter Weibull distributions. For each data set, the OSL number for pointed distributions were calculated and listed in Tab 4.

Tab. 4: Results of Anderson-Darling goodness-of-fit test.

Data set	OSL Normal distribution [-]	OSL Lognormal distribution [-]	OSL Weibull distribution [-]
0° fibre orientation	0.5014	0.5423	0.1447
90° fibre orientation	0.6527	0.6510	0.6550
45° fibre orientation	0.4146	0.4391	0.1724

Based on results listed in Tab. 4, it can be concluded that any of the distributions are able to approximate prescribed set of data. Therefore, the B-values for each distribution were calculated. These values are listed in Tab. 5

Tab. 5: Calculated B-values.

Data set	B-value according to Normal dist. [MPa]	B-value according to Lognormal dist. [MPa]	B-value according to Weibull dist. [MPa]
0° fibre orientation	122630.0	123067.6	110168.7
90° fibre orientation	7363.0	7366.4	7130.6
45° fibre orientation	12145.0	12160.2	11443.1

Conclusion

Stiffness and strength parameters of the manufactured composite were determined and statistically evaluated. One sample was detected as an outlier from the strength point of view. Corresponding samples from two batches were combined into sets of data and then goodness-of-fit tests were performed. Unfortunately it was concluded that the data sets cannot be approximated with any of the listed distributions and therefore B-values were calculated. For future purposes, more sample batches will be manufactured and measured.

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