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Calculation of B-Basis Values from Composite Material Strength Parameters Obtained from Measurements of Non-Identical Batches

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Abstract. Experimental investigation of stiffness and strength parameters of unidirectional carbon fibre plates produced in an autoclave using the Vacuum Assisted Process was performed. Parameters belonging to five production batches were subjected to the statistical evaluation. The Maximum Normed Residual test was used for the identification of outliers and the k-sample Anderson-Darling test was used in order to determine whether the parameters can be processed as belonging to one big group of data. Due to the previous work and negative result of the test for strength parameters, the scope of the work was focused to the evaluation B-basis values using the Analysis of variance method.

Introduction

High stiffness and strength to weight ratios of composite materials cause increasing use of these materials in various applications in sport, automotive, marine, and aerospace industries. However, manufacturing process is a very complex process consisting of a fiber placement done by a worker or a robot, a potential resin infusion and from a curing process. These process critical points may lead to batch-to-batch variability caused by process variability. Except of listed process critical points, also raw materials batch-to-batch variability and testing variability may lead to a final material variability [1]. This material variability results in stiffness and strength variability of a final composite product. Therefore, it is essential to use a statistical evaluation of experimentally investigated material parameters. These parameters should be obtained from measurements of samples belonging to different batches and later the hypothesis, whether it can be processed as belonging to one group, should be tested.

In case of the positive test, a statistical distribution can be usually used for the approximation. In [1], the Normal distribution, the Lognormal distribution and the 2-parameter Weibull distribution are recommended as the most suitable for the approximation of material parameters of composites. The Normal distribution was used in [2] for the approximation of the stiffness and strength parameters of carbon fibre composite. The Weibull distribution was used in [3], [4], and [5]. These three papers were focused on the approximation of the strength parameters of unidirectional carbon fibre samples, of carbon/epoxy laminate having the layup $[0/90]_{2S}$, and of bamboo fibres, respectively. However, none of the papers mention whether the material parameters were measured using samples from one or more batches. Therefore, the test for the hypothesis, whether the parameters can be processed as belonging to one group, was not mentioned. Due to the previous work of the author [6], it was observed that the material strength parameters tend not to confirm the hypothesis of behaving as belonging to one group (non-identical batches).

Therefore, this work was focused on the extension of the previous work in form of the batch count increase and on the evaluation of B-basis values from material strength parameters measured from non-identical batches.

Theoretical background

The 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:

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

where $\bar{x}$ denotes the sample mean and s 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 the 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].

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

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

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

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

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

The One-way Analysis of variance

The one-way Analysis of variance (ANOVA) can be used for the calculation of the B-basis value in case of negative result of the k-sample Anderson-Darling test. The analysis can be used only if following assumptions are fulfilled [1]:

1. The data from each batch are normally distributed,
2. The within-batch variance is the same from batch to batch,
3. The batch means are normally distributed.

Currently, there is no available test for the first assumption. For the second assumption, there is advised to use the Levene's test for equality of variances. Also for the third assumption, there is no useful test if the batch number is not sufficient high (twenty or higher) [1].

For the calculation of the B-basis value, following calculations must be performed:

$$n^* = \sum_{i=1}^{k} n_i^2/n, \tag{10}$$

$$n' = (n - n^*)/(k-1), \tag{11}$$

$$\bar{x} = \sum_{i=1}^{k} n_i \bar{x}_i/n, \tag{12}$$

$$MSB = \sum_{i=1}^{k} \frac{n_i(\bar{x}_i - \bar{x})^2}{k-1}, \tag{13}$$

$$MSE = \frac{1}{n-k} \sum_{i=1}^{k} (n-k) s_i^2, \tag{14}$$

where n_i is the number of samples in i-th batch, n denotes the total number of samples across k batches. n' is the effective batch size, $\bar{x}_i$ and s_i are the batch mean and batch standard deviation, respectively. $\bar{x}$ denotes the overall mean. MSB is called between-batch mean square and the MSE is called the within-batch mean square.

Furthermore, it is necessary to calculate the population standard deviation:

$$S = \sqrt{\frac{MSB}{n'} + \left(\frac{n'-1}{n'}\right) MSE}, \tag{15}$$

and also the ratio of mean squares defined as:

$$u = \frac{MSB}{MSE}. \tag{16}$$

The tolerance limit factor can be calculated as follows:

$$T = \frac{k_0 - \frac{k_1}{\sqrt{n'}} + (k_1 - k_0)w}{1 - \frac{1}{\sqrt{n'}}},$$

where

$$w = \sqrt{\frac{u}{u + n' - 1}} \tag{17}$$

and parameters k_0 and k_1 are tolerance limit factors for a simple random sample from a normal distribution with sample size *n* and size *k*, respectively. Mentioned parameters can be found in tables [1].

Thus, B-basis value can be calculated using formula:

$$B = \bar{x} - TS. \tag{18}$$

The Levene's test for equality of variances

One of the assumptions for the applicability of the ANOVA method is the equality of batch variances. For this purpose, the Levene's test is widely used. For this test, data must be transformed as follows:

$$w_{ij} = \left| x_{ij} - \tilde{x}_i \right|, \tag{19}$$

where $\tilde{x}_i$ is the median of the n_i values of the *i*-th batch. Using transformed data, the F-test must be performed. If the test statistics is greater than or equal to the tabulated F-distribution quantile, the variances are significantly different [1].

The F-test

The F-test is used for testing of batch mean (or variance) equality. In order to test the hypothesis if the sample batches have the same mean, the statistics must be calculated [1]:

$$F = \frac{\sum_{i=1}^{k} n_i (\bar{x}_i - \bar{x})^2 / (k-1)}{\sum_{i=1}^{k} \sum_{j=1}^{n_i} \left(x_{ij} - \bar{x}_i\right)^2 / (n-k)} \tag{20}$$

where $\bar{x}_i$ is the mean of n_i values in the i-th group and $\bar{x}$ is the mean of all n observations. If the F statistics is greater than $1 - \alpha$ quantile of the F-distribution, it can be concluded (with α percent risk of error), that the means of *k* groups are not equal.

Test Samples

For purposes of the test sample creation, five carbon fibre plates with unidirectional (UD) fibre orientation were produced by the author in the ASC Econoclave autoclave (Fig. 1). Plates were produced from non-crimped fabric (NCF) Saertex with fibres TENAX-J IMS60 E13 24K and from epoxy resin MSG L285 using the Vacuum Assisted Process (VAP®). VAP® is the process patented the Airbus Group and it is using the VAP® membrane made out of gore-tex®. This membrane is used for separation of the air (vacuum) from the resin. Thus, the amount of air bubbles contained in resin is reduced and nearly optimal fibre to resin ratio is achieved.

Test samples were cut from produced plates using water jet cutter. Samples for testing of tensile and shear material parameters were cut from each plate. Therefore, obtained samples had fibre orientation 0°, 45° and 90° with respect to the longer edge of a sample. The dimensions followed recommendations of ASTM D3039 and were determined as 200 mm × 25 mm × 2.4 mm (Fig. 2). Six samples per each batch and fibre orientation were cut and numbered to guaranty the full traceability.

All samples were measured and weighted for purposes of the fibre volume ratio calculation. Resulting means and standard deviations of the fibre volume ratios for each batch are listed in Table 1.

Table 1: Mean fibre volume ratios and standard deviations of tested batches.

Batches	Mean fibre volume ratio [%]	Standard deviation of fibre volume ratio [%]
1	60.7	0.9
2	64.0	0.9
3	61.6	0.7
4	61.6	0.5
5	62.6	0.4

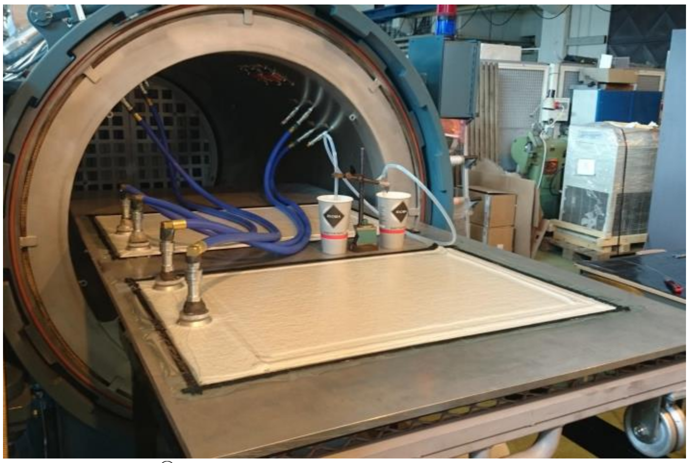

Fig. 1: VAP® manufacturing process of UD carbon fibre plates.

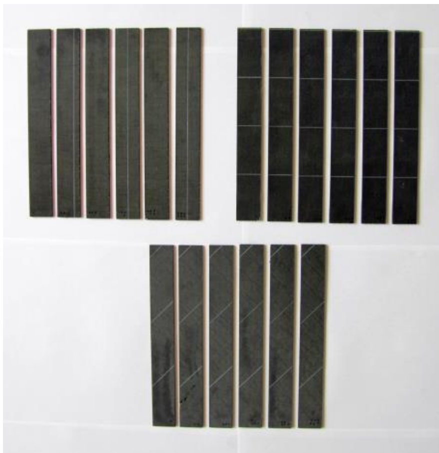

Fig. 2: Example of sample batch with fibre orientation 0°, 90° and 45°.

Experiments and statistical evaluation

Experiments were carried out using the universal testing machine Zwick/Roell Z050. The loading rate of 2 mm/min was selected based on recommendation of the ASTM D3039. The load was measured using a load cell and the displacement of the sample was measured using an extensometer. The gage length was set to 60 mm. All samples were meant to load up to the failure.

However, it was observed that the load and also the clamp capability of the testing machine were not sufficient for the test samples having the fibre orientation of 0°. Therefore, it was not possible to measure properly the stiffness and strength parameters of these samples and they were removed from the evaluation.

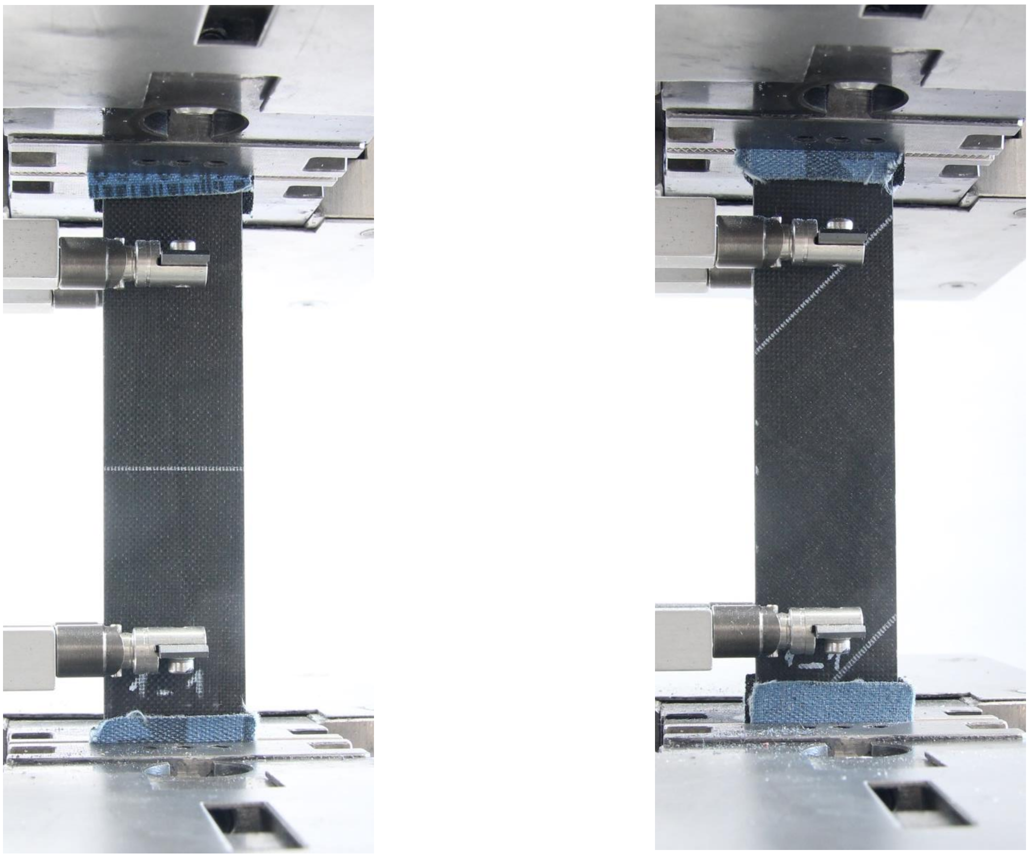

Fig. 3: Experiments with samples having 90° (left) and 45° (right) fibre orientations.

Obtained load-displacement curves were recalculated into the engineering stress-strain curves and stiffness and strength parameters were evaluated. These parameters were subjected to the MNR test. Based on the results, sample no. 2 having the 45° fibre orientation from the batch 2 and sample no. 3 having the 45° fibre orientation from the batch 5 were detected as outliers from the strength point of view. Therefore, they were removed from other evaluations. Without mentioned outliers, all batches passed the MNR test. Resulting critical values are listed in Table 2 and Table 3. Based on the parameters for each sample and batch, mean values and standard deviations were calculated for stiffness and strength parameters. Parameters related to the samples having fibre orientation of 90° and 45° are listed in Table 4 and Table 5, respectively.

Table 2: MNR and CV values for stiffness and strength parameters for samples having 90° fibre orientation

Batch	MNR for stiffness parameters [-]	CV for stiffness parameters [-]	MNR for strength parameters [-]	CV for strength parameters [-]
1	1.594	1.887	1.484	1.887
2	1.239	1.887	1.310	1.887
3	1.271	1.887	1.314	1.887
4	1.620	1.887	1.491	1.887
5	1.415	1.887	1.534	1.887

Table 3: MNR and CV values for stiffness and strength parameters for samples having 45° fibre orientation

Batch	MNR for stiffness parameters [-]	CV for stiffness parameters [-]	MNR for strength parameters [-]	CV for strength parameters [-]
1	1.586	1.887	1.514	1.715
2	1.517	1.887	1.720	1.887
3	1.881	1.887	1.591	1.887
4	1.305	1.887	1.676	1.887
5	1.717	1.887	1.200	1.715

Table 4: Mean values and standard deviations of stiffness and strength parameters for samples having 90° fibre orientation

Batch	Stiffness mean [MPa]	Stiffness standard deviation [MPa]	Strength mean [MPa]	Strength standard deviation [MPa]
1	7685.2	114.2	43.1	1.0
2	7602.8	148.4	39.7	0.9
3	7822.3	244.7	40.4	1.2
4	7760.8	238.0	40.7	1.4
5	7946.1	126.6	41.9	1.5

Table 5: Mean values and standard deviations of stiffness and strength parameters for samples having 45° fibre orientation.

Batch	Stiffness mean [MPa]	Stiffness standard deviation [MPa]	Strength mean [MPa]	Strength standard deviation [MPa]
1	11247.3	401.6	67.0	2.5
2	11474.8	537.7	62.5	1.9
3	10875.4	561.0	63.1	2.5
4	11814.8	887.0	63.3	1.9
5	11568.1	357.6	64.7	2.0

Furthermore, parameters from all batches were subjected to the k-sample Anderson-Darling test in order to test the hypothesis whether parameters can be processed as belonging to one big group of samples (further referred to as pooled parameters). This hypothesis was confirmed for the stiffness parameters for samples having the fibre orientation of 90° and also of 45° (Table 6). The further evaluation of pooled parameters was the scope of the previous work and it is described in [6]. However, for the strength parameters, this hypothesis was rejected. Therefore, it was necessary to perform the Levene's test for equality of variances, which is involving the F-test, in order to test whether one of the requirements for the application of ANOVA method for calculation of B-basis values is fulfilled (Table 7). Listed critical value is the 0.95 quantile of the F-distribution.

Table 6: Results for k-sample Anderson Darling test.

Samples	ADK for stiffness param. [-]	ADC for stiffness param. [-]	ADK for strength param. [-]	ADC for strength param. [-]
90° fibre orientation	1.6123	1.6412	2.4490	1.6412
45° fibre orientation	1.5896	1.6412	2.5584	1.6356

Table 7: Results for the Levene's test for equality of variances

Sample strength parameters	F [-]	Critical value [-]
90° fibre orientation	0.17	2.76
45° fibre orientation	0.30	2.80

Based on the results from the Table 7, it was concluded that the ANOVA method can be used for the calculation of B-basis values for the strength parameters. Using the eq. 10 through eq. 18, B-basis values and population standard deviations were calculated and listed in the Table 8.

Table 8: B-basis values and population standard deviations for the strength parameters

Sample strength parameters	B-basis value [MPa]	Population standard deviation [MPa]
90° fibre orientation	36.3	1.7
45° fibre orientation	57.4	2.6

Conclusion

The statistical evaluation of the strength material parameters of the produced carbon fibre plates was performed. Two outliers from the strength point of view were removed from batches and the B-basis values were calculated in case of the negative k-sample Anderson-Darling test. The investigation of measurement possibilities for the high strength samples having the 0° fibre orientation, the evaluation of the B-basis values for these samples and the use of calculated values for the research of bolted joint strength will be the scope of the future work.

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