

# STATISTICAL ANALYSIS OF EXPERIMENTAL RESULTS IN THE IN-PLANE SHEAR TEST FOR A CROSS-PLY VINYLESTER-CARBON LAMINATE

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## **Abstract**

*The study presents experimental studies on a regular cross-ply laminate of a  $[(0/90)_F]_{4S}$  configuration. Each layer is made of VE 11-M vinylester resin (the manufacturer: „Organika-Sarzyna” Chemical Plants) reinforced with a plain carbon fabric (the manufacturer: C. Cramer GmbH & Co. KG Division ECC). The carbon fabric has the following parameters: Styles 430, filament Carbon 6K, substance 300 g/m<sup>2</sup>, warp and weft 400/400 tex, 3.7/3.7 band/cm. Laminate was produced by ROMA private enterprise using the vacuum molding technology and technological parameters developed by ROMA taking into account the VE 11-M material specification.*

*A program of the experimental studies was aimed on statistical analysis of measurement results of the in-plane shear modulus and the in-plane shear strength of the laminate. In investigations, the in-plane shear test was carried out in accordance to the PN-EN ISO 14129:1997 standard [1], but applying the modified procedure that takes into consideration the results of the previous research on the influence of a traverse velocity and of a number of stress cycles on static measurements of the in-plane shear modulus [2].*

*The mean value, the standard deviation and the two-sided 95% confidence interval for the mean value for the investigated quantities have been determined. Practical usefulness of the manufacture technology of the laminate was assessed.*

**Keywords:** *cross-ply laminate, vinylester-carbon laminate, in-plane shear modulus, in-plane shear strength, experimental studies, statistical analysis*

## **1. Introduction**

The results of the experimental tests show some dispersion of obtained values, also observed in testing the mechanical properties of composites panels. Especially, their mechanical properties can vary significantly due to micro-heterogeneity of composite panels and to fragility of matrix and fibers. Variability of the results may be also caused by errors of the measurement system [3]. All these factors point that statistical methods for processing of the experimental results are necessary, allowing determining a distribution of the variable and its parameters [4]. In the literature, the Weibull, the Gauss, the t-Student, and  $\chi^2$  distributions are applied to describe the studied mechanical properties [5, 6].

Statistical analysis of the results for polymer-matrix composites is standardized by the ISO 2602:1994 standard [4]. Models of statistical processing and analysis of the results, which are listed in this standard, make possible determining the interval with a given probability which contains the average of the total population made up of the results of a number of measurements carried out in the same conditions. Standard [4] describes conditions for the appointment of the confidence interval for the mean. The confidence interval is related to the level of confidence (sometimes called a confidence coefficient) which is understood as probability of covering the population by the interval, expressed as a percentage.

The study presents statistical analysis of the results of the measurements related to the in-plane shear test performed by a tensile test of a  $[(\pm 45)_F]_{4S}$  regular cross-ply laminate, denoted with the C/VE code, which is described in detail in further considerations. This test for polymer-matrix composites is one of the most difficult tests for determining the mechanical properties of laminates. The measurement results were preceded statistically according to ISO 14129:2000 [1] or its American counterpart, i.e. ASTM D3518/D3518M-94 [7].

The in-plane shear test allows for determining shear stress  $\tau$  and shear strain  $\gamma$  in orthotropic polymer-matrix composites of high ultimate strains. This method is used to determine the  $G_{12}$  in-plane shear modulus based on the initial straight section of the  $\tau$ - $\gamma$  relation, as well as to determine the  $R_{12}$  in-plane shear strength. The failure criterion is related to the shear stress corresponding to the 5% ultimate shear strain if break of the specimen does not occur below this value.

The studies presented in this paper have the following objectives:

- a) statistical analysis of the experimental results for  $G_{12}$ ,  $R_{12}$  using the modified experimental procedure developed in Ref. [2],
- b) assessment of practical usefulness, as a structural material, of a cross-ply C/VE laminate produced by ROMA enterprise using the vacuum molding technology.

## 2. The theoretical basis

Statistical analysis, through the use of the defined metrics and the graphical methods, allows interpreting the experimental results by determination of their distribution, arithmetic mean, standard deviation and confidence interval of mean value for the t-student distribution [1, 3, 4]. The basic statistical value is the arithmetic mean of the results [1, 4]:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i . \quad (1)$$

An average value calculated for a limited number of specimens is different from the mean value for the whole population, but it tends to the latter as a number of specimens increases [3].

A measure of dispersion of the results, assuming the normal distribution, is the standard deviation calculated as [1, 3, 4]:

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2} . \quad (2)$$

Formula (2) results from the limited set of multiplicity of the results obtained, and the substitution of the  $n-1$  instead of  $n$  allows increasing the accuracy [3].

Another statistical characteristic which is used to interpret the results is two-sided 95% confidence interval for the mean value, i.e.:

$$\bar{x} - \frac{t_{0,975}}{\sqrt{n}} s < m < \bar{x} + \frac{t_{0,975}}{\sqrt{n}} s , \quad (3)$$

where:

$\bar{x}$  - arithmetic mean of  $n$  measurements,

$n$  - number of all measurements,

$x_i$  - value of the  $i$ -measurement,

$s$  - standard deviation,

$m$  - mean value for the population.

The value of  $t_{0,975}$ , which has to be used to calculate the two-sided confidence interval with a given probability, is determined from the t-student distribution of  $v = n-1$  degrees of freedom. The value of this characteristic is given in the standard [4]. The shear strength  $R_{12}$  and the shear modulus

$G_{12}$ , which values were used in statistical analysis, were calculated with the three digits accuracy.

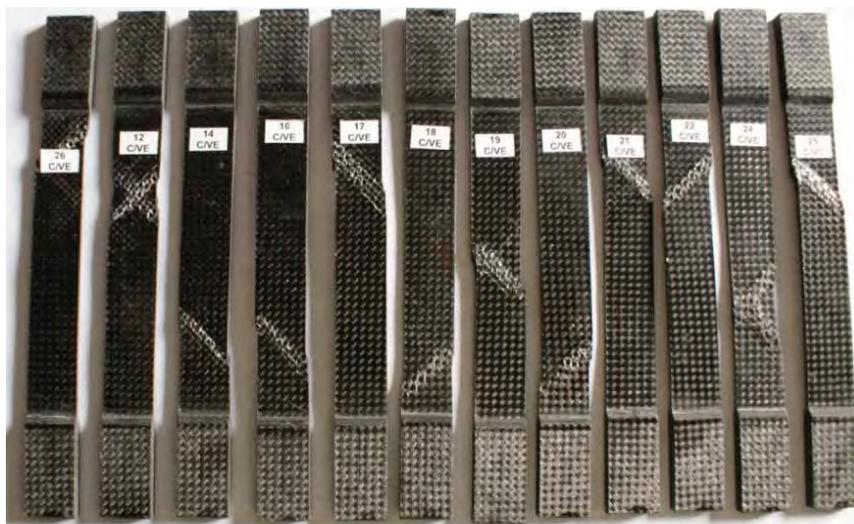
Statistical analysis and graphical presentations of the results allow to properly interpreting the test results. The following comments were taken into account during the statistical analysis:

- 1) The normal distribution is the default probability distribution adopted as a mathematical model of probability distribution for the population in the experimental studies. For such distribution, two parameters –the mean value  $m$  and the standard deviation  $s$  – are unknown. In most cases, the assumption of normality is satisfied, since the distribution of measurement results is a normal distribution or a distribution close to its, for fixed terms of data collection. It is desirable to check whether this assumption can be adopted.
- 2) Calculations can be simplified by shifting the origin or changing the unit of measurement. However, it is dangerous to round of the numerical results of the measurements. It is unacceptable to reject any observation or application of any amendments to the observation of clearly dubious, without any justification based on experimental, technical or other considerations that raise no objection.
- 3) The test method may be affected by systematic errors. The occurrence of such errors may prevent the correct use of the presented method. In some cases, the methods described in ISO 2854 [8] may be useful to identify systematic errors. Research conducted in the context of this work was preceded by calibration and verification tests that eliminate possibility of systematic errors.

### **3. The experimental results and their statistical analysis**

According to the modified procedure described in Ref. [2], the in-plane shear test was carried out in two cycles, for 12 specimens, using the velocity of the traverse motion of the universal testing machine equal to  $v = 2$  mm/min (control movement). The first cycle of the control movement was of the triangular distribution, with the maximum shaping strain of  $\gamma \leq 0.0050$ , while the next one was carried out for  $v = \text{const}$ , until the breaking of the specimen appears (Fig. 1). The first cycle is an introductory cycle inducing redistribution of the manufacture residual stresses in the form of quasi-uniformly distributed microcracks in the whole laminate volume. The second cycle is a measurement cycle for determining the investigated material constants.

The experiments have shown three forms of damage mechanisms of the C/VE cross-ply laminate in the test undertaken shown in Fig. 1.



*Fig. 1. A photo of the 12 damaged specimens in the in-plane shear test*

### 3.1. The tested material

The test specimens were made from the composite prefabricated plate products, cut to obtain the  $[(\pm 45)_F]_{4S}$  configuration. The plates were manufactured as a stack of layers reinforced by an orthogonal fabric of a simple interlacing. During the test, the specimen in the shape of a rectangular prism with the fibers oriented at the angles of  $\pm 45^\circ$  to the longitudinal axis of the specimen in each stratum was subjected to the respective unidirectional tensioning load. The specimen material during the experiment is not in the pure shear state, since both the shear and normal stresses appear in the system  $\pm 45^\circ$  (Fig. 2).

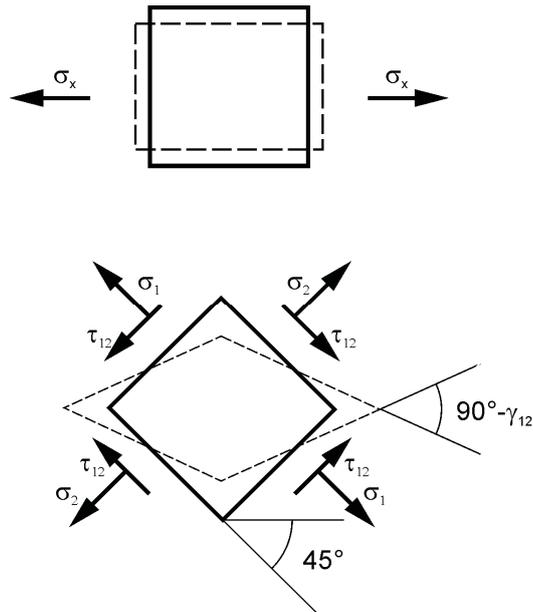


Fig. 2. The planar stress state in the in-plane shear test

The previously conducted studies [3, 9, 10] show that the normal stresses have a negligible effect on the value of the in-plane shear modulus,  $G_{12}$ , and slightly decrease the in-plane shear strength,  $R_{12}$ .

The regular cross-ply C/VE laminate of the  $[(0/90)_F]_{4S}$  configuration was considered. Style 430 fabric was produced by the ECC using Tenax-E HTA40 carbon roving bands. The fabric was characterized by the following parameters: Style 430, a simple weave, thickness 0.42 mm, width 1000 mm, substance  $300 \text{ g/m}^2$ , warp/tex thread 400/400, 3.7/3.7 bands/cm. The VE 11-M vinylester matrix – applied to obtain the laminate with increased requirements for chemical resistance and fireproof – was used to manufacture the tested laminate.

Prefabricated plate products were produced by ROMA enterprise using the vacuum molding technology with the technological parameters developed by ROMA taking into account the VE 11-M resin card provided by the producer.

### 3.2. The results of experimental studies

For each specimen, satisfying the requirements of the standard dimensional tolerances, the in-plane shear modulus,  $G_{12}$ , according to the standard and modified procedures [1, 2] and the in-plane shear strength,  $R_{12}$ , according to the modified procedure, were calculated. All specimens were destroyed in the area of permissible destruction. The results obtained are provided in Tab. 1.

Tab. 1. The results of the in-plane shear test

specimen No.	specimen code	$G_{12}$ [GPa]		$R_{12}$ [MPa]	$\gamma$
		the procedure from PN-ISO 14129 [1]	the modified procedure [2]		
1	P12	4.37	4.78	47.7	0.0386
2	P14	4.36	4.67	48.8	0.0486
3	P16	4.25	4.45	43.2	>0.0500
4	P17	4.27	4.47	43.0	>0.0500
5	P18	4.45	4.55	46.3	>0.0500
6	P19	4.36	4.69	45.8	>0.0500
7	P20	4.13	4.29	47.0	>0.0500
8	P21	4.37	4.77	46.0	>0.0500
9	P22	4.53	4.89	47.7	0.0271
10	P24	4.23	4.71	46.9	>0.0500
11	P25	4.15	4.39	44.5	>0.0500
12	P26	4.18	4.33	46.2	>0.0500

The specimens have showed two types of the behaviour: either the shear strain had reached the ultimate value of 0,0500 with the stress increased monotonically (Fig. 3), or it had reached this value not monotonically or they had had the stress drop (Fig. 4).

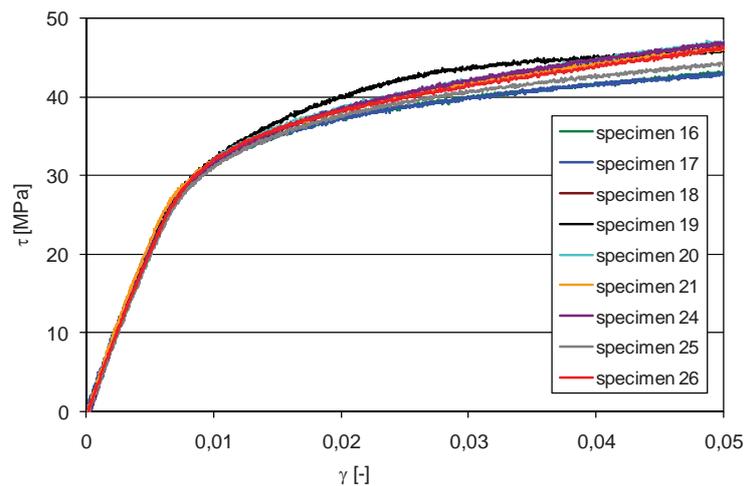


Fig. 3. The specimens which have reached the value of shear strain  $\gamma = 0.0500$  with the stress increasing monotonically (cycle 2)

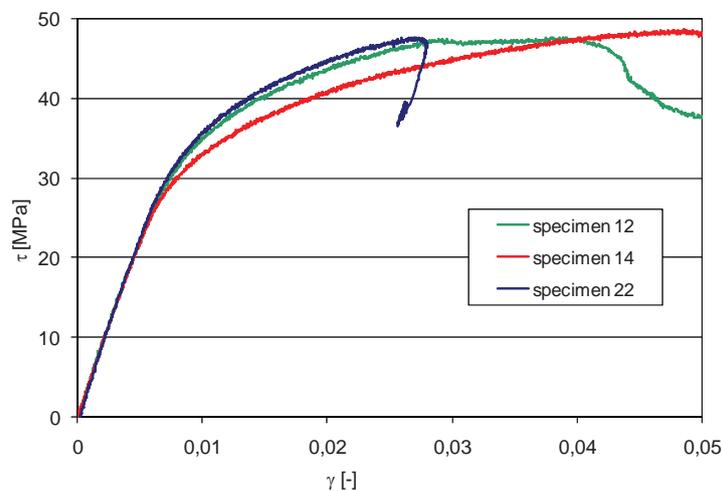


Fig. 4. The specimens which have been destroyed for  $\gamma < 0.0500$  in cycle 2 (specimen 22)

The linear regression method was used to determine the in-plane shear modulus. Its accuracy is determined by the number of points that enter into calculations, so it should use a sufficiently large sampling frequency.

The  $\tau$ - $\gamma$  relation for each specimen was used to determine the module with the original  $G_{12}$  cutting section of the graph using a simple linear regression for both procedures (Fig. 5, 6).

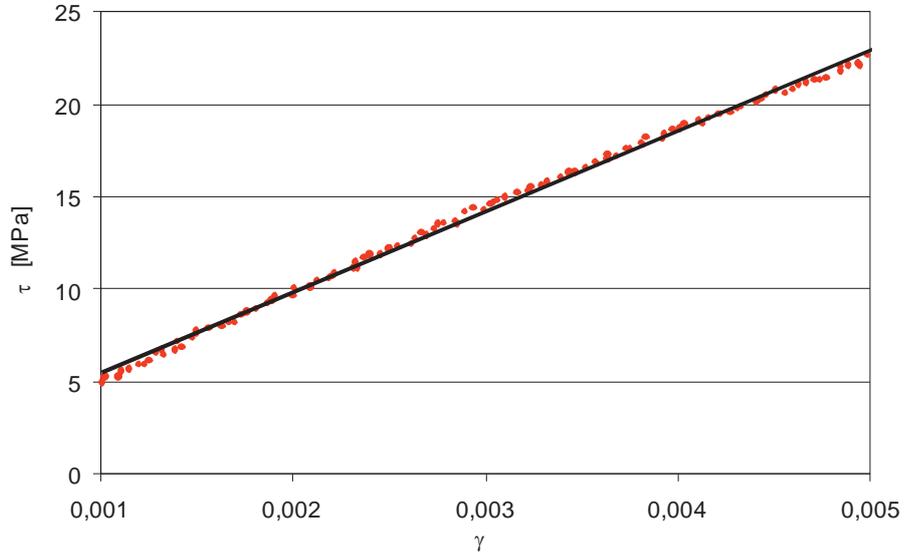


Fig. 5. The  $\tau$ - $\gamma$  diagram for  $v = 2$  mm/min (specimen 12, cycle 1), according to the ISO 14129 procedure [1]

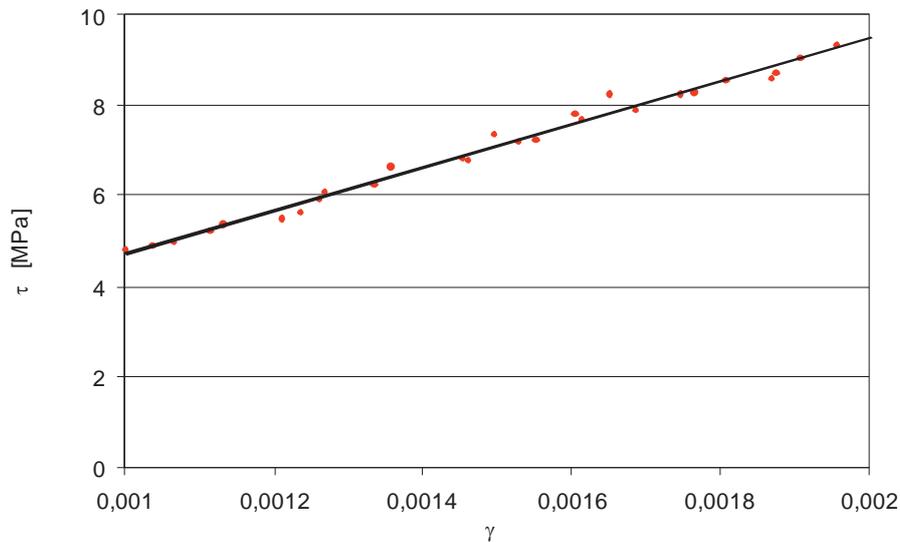


Fig. 6. The  $\tau$ - $\gamma$  diagram for  $v = 10$  mm/min (specimen 12, cycle 1), according to the modified procedure [2]

### 3.3. Statistical analysis of experimental results

The results of statistical calculations in accordance to Eqs. (1, 2, 3) are summarized in Tab. 2, assuming  $\nu = n-1 = 11$  for the two-sided intervals and the following data taken from the tables contained in the PN ISO 2602:1994 standard [4]:

$$t_{0,975} = 2.201, \quad \frac{t_{0,975}}{\sqrt{n}} = 0.635. \quad (4)$$

Tab. 2. The results of statistical calculations of the material properties of the C/VE laminate

Data type	$G_{12}$ [GPa]		$R_{12}$ [MPa]
	the procedure form PN-ISO 14129 [1]	the modified procedure [2]	
$\bar{x}$	4.30	4.58	46.09
s	0.12	0.20	1.77
$\frac{t_{0,975}}{\sqrt{n}} \cdot s$	0.08	0.12	1.13
$x_d$	4.22	4.46	44.96
$x_g$	4.38	4.70	47.22

$x_d$  - lower confidence limit for the arithmetic mean for the whole population,  
 $x_g$  - upper confidence limit for the arithmetic mean for the whole population.

For graphic interpretation of the test results, grouping of the frequency distribution has been used, i.e., a histogram. In this way, the probability distribution can be verified [3].

Overview of the in-plane shear module obtained for both procedures shows that the values obtained from the modified procedure are always higher than the values obtained by the standard procedure (Fig. 7).

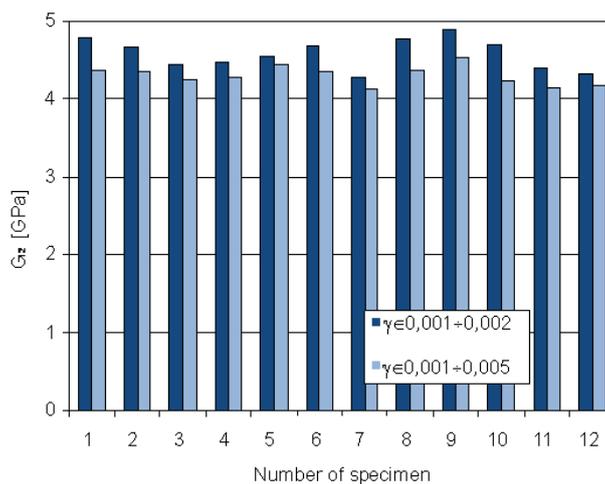


Fig. 7. Overview of the  $G_{12}$  shear modulus obtained from both tested procedures

Shear strength values of  $R_{12}$ , determined for examined test specimens, belong to the 40-50 MPa interval (Fig. 8).

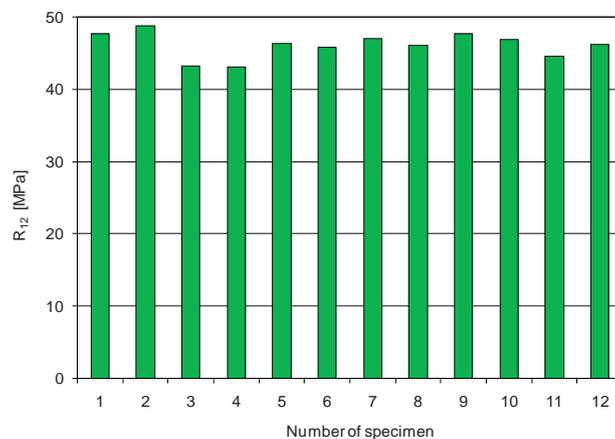


Fig. 8. Summary of the  $R_{12}$  shear strength

Calculated values for the shear modulus  $G_{12}$ , and the shear strength  $R_{12}$  are reflected in the form of histograms showing the nature of the probability distribution of the tested quantities (Fig. 9-11). The probability distribution function for the shear modulus  $G_{12}$  from the modified procedure is close to the normal distribution (Fig. 9). For the standard procedure [1] (Fig. 10) one cannot observe this feature. The probability distribution function for shear strength  $R_{12}$  (Fig. 11), determined from the modified procedure [2], is also close to the normal distribution.

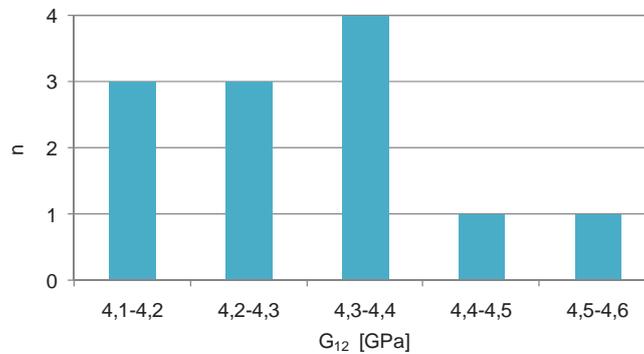


Fig. 9. A histogram of the  $G_{12}$  shear modulus based on the modified procedure [2]

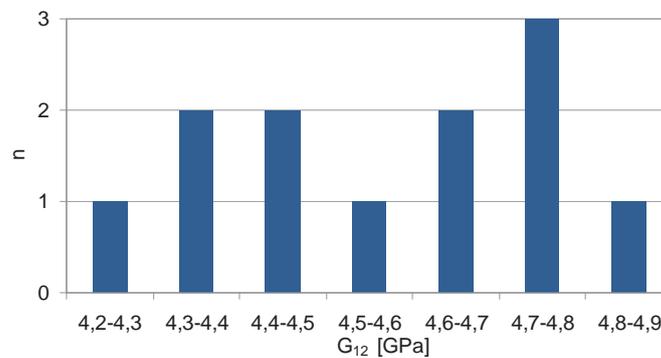


Fig. 10. A histogram of the  $G_{12}$  shear modulus obtained using the ISO 14129 procedure [1]

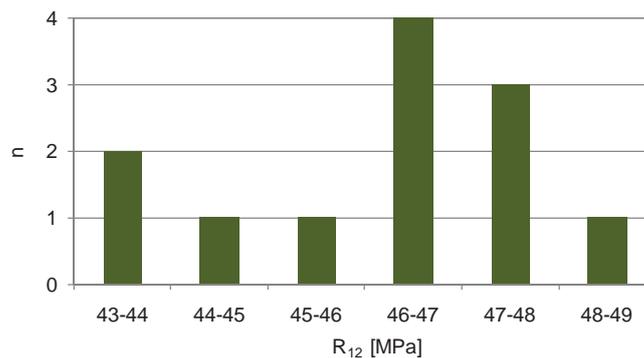


Fig. 11. A histogram of the  $R_{12}$  shear strength obtained using the modified procedure [2]

#### 4. Final conclusions

Based on statistical analysis of the measurements results in the in-plane shear test, the following final conclusions can be formulated:

- 1) Three failure mechanisms for a cross-ply C/VE laminate in the test undertaken have been identified. All specimens were destroyed in the area of permissible destruction. Shapes of failure mechanisms and zones of destruction constitute evidence for correctness of the

manufacture technology and practical usefulness of the test method.

- 2) If the specimen was destroyed outside the extensometer measurement region, as shown in Fig. 3, it does not reach the value of  $\gamma = 0.0500$ .
- 3) The values of the  $G_{12}$  in-plane shear modulus calculated from the modified procedure are higher than respective values calculated from the standard procedure by 6.4% (the average value).
- 4) A histogram of the  $G_{12}$  shear modulus obtained from the normalized procedure is significantly different from the normal distribution. It demonstrates the validity of a modified procedure.
- 5) Statistical parameters corresponding to the constants  $R_{12}$  and  $G_{12}$  confirm practical usefulness of the cross-ply C/VE laminate produced by the ROMA enterprise with the vacuum moulding technology, for the use as a structural material. The laminate can be considered as a homogeneous material in a macroscale.

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