

# INFLUENCE OF A CROSSHEAD RATE AND A NUMBER OF STRESS CYCLES ON MEASUREMENT RESULTS IN THE IN-PLANE SHEAR TEST FOR A CROSS-PLY VINYLESTER-CARBON LAMINATE

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## Abstract

The paper presents some experimental studies on a regular cross-ply laminate of the  $[(0/90)_F]_{4S}$  configuration. Each layer is VE 11-M vinylester resin (the manufacturer: „Organika-Sarzyna” Chemical Plants, Sarzyna, Poland) reinforced with pla in weave carbon fabric of parameters: S tyle 430, Carbon 6K, substance  $300 \text{ g/m}^2$ , warp/weft 400/400 tex, 3.7/ 3.7 yarn/cm (the manufacturer: C. Cramer GmbH & Co. KG Division ECC). The orthotropic laminate was produced by ROMA private enterprise in Grabowiec, Poland, using the vacuum molding method and the technological parameters developed by ROMA taking into account the VE 11-M material specification.

The PN-EN ISO 14129:1997 standard [1] and closely related standards [2-9] were taken into consideration in experimental studies on the static in-plane shear response by a tensile test of a  $[(\pm 45)_F]_{nS}$  laminate. A program of the experiments was focused on testing a rate of a testing machine crosshead and a number of static stress cycles. An influence of these factors on the in-plane shear modulus was investigated.

Based on the conducted investigations, the modified experimental procedure has been proposed for determination of the correct value of the in-plane shear modulus and the in-plane shear strength.

**Keywords:** vinylester-carbon laminate, regular cross-ply laminate, , in-plane shear test, experimental procedure

## 1. Introduction

Shear tests for polymer-matrix composite materials are the most difficult tests for determining mechanical properties, particularly in the plane perpendicular to the laminate plane. There are well-known numerous methods for shear tests of laminates. The most important of them are described below [10-13].

Two or three rails, which the specimens are attached to, are applied in the method of shear test in rails (ASTM D4255/D4255M-01 [2]). This method defines the in-plane shear modulus and the in-plane shear strength. The length/width ratio for the measured region of the specimen is recommended to be greater than 10. The Iosipescu test method (ASTM D5379/D5379M-05 [3]) uses four-point bending of the notched prismatic specimen. It allows determining the mechanical properties in all planes. Both methods require special instrumentation.

The next method of a shear test applies a tensile test of a  $[(\pm 45)_F]_{nS}$  laminate (ISO 14129:2000 [1] or – its American equivalent – ASTM D3518/D3518M-94 [4]). This method allows determining the shear stress,  $\tau$ , and the shear strain,  $\gamma$ , for polymer-matrix composites with large failure deformations. It can be also used to determine the value of the in-plane shear modulus,  $G_{12}$ , reliable since it is based on the straight section of the initial  $\tau$ - $\gamma$  relation.

The plate torsion test (PN-EN ISO 15310:2007 [5]) allows only determining the in-plane shear modulus ( $G_{12}$ ). The test uses simultaneous loading of the plate supported at two corners along its diagonal with cones placed at corners along the other diagonal.

Another method is a torsion test of a rod with circular or rectangular cross-section (PN-C-89076 [6]), with or without a hole. The method, for determining the apparent interlaminar shear strength of fiber-reinforced composites using a short beam (PN-EN ISO 14130:2001 [7]), is applied to laminates with thermoset or thermoplastic matrix, under condition of damage caused by the interlaminar shear. This method can be applied to classify or to control the quality the materials. Standards related to the interlaminar shear strength of the plastics, developed separately for aviation and cosmonautics, are as follows: PN-EN 2377:1994 [8] – for glass fiber-reinforced composites, and PN-EN 2563:2000 [9] – for unidirectional carbon fiber-reinforced composites.

Besides the above-mentioned methods, there exist the others, such as: the tube twisting method (this method gives the most reliable results), the shear test using the four-bar linkage (the in-plane shear; this method gives correct results for the in-plane shear strength, but it is not suitable for determining the shear modulus), shear of a specimen in biaxial load (applied for many materials), torsion of ring specimens (applied in investigation of a pipe-shaped laminate), shear of a specimen with notches (however, resulting in a large error) [12]. Originally, many of these test methods were dedicated to materials different from composites reinforced with continuous fibers (metals, wood, polymers). Some of them are not even standardized.

Compatibility of these methods related to accuracy of determining the shear modulus is noted, whereas determining of the shear strength is more problematical. The edge effects, connecting of materials, non-linear behaviour of the matrix or the matrix-fibers connection, stress distribution (imperfections), existing of the normal stresses, cause that the value of the shear strength determined by existing methods is disputable. Therefore, the value of shear strength in engineering applications should be checked for each structure.

Some of the test methods were proposed to determine the  $\tau$ - $\gamma$  relation in a plane of the layer for fiber-reinforced composites, the other ones are confined to the plane perpendicular to the previous one and some – in dependence of cut of the specimen – allow researching both cases.

Summing up, the shear modulus and/or the shear strength are determined on the basis of the shear test. It is also possible to determine the failure strain.

## 2. Description of the experimental studies

The aim of this study is to determine the influence of selected factors, i.e., a rate of the testing machine crosshead, a number of cycles of static stress, and sensors used for strain measurement, on the shear stress – shear strain relation in the in-plane shear test [1]. There is considered a vinylester-carbon regular cross-ply laminate denoted with the C/VE code.

The modified procedure will be developed related to the test method presented in PN-EN ISO 14129 standard [1]. It results from the fact that this standard does not take into account above-mentioned factors.

### 2.1. Description of the test method

The shear test method corresponds to the PN-EN ISO 14129 standard [1] and permits to determine the shear stress,  $\tau$ , and the shear strain,  $\gamma$ , of polymer-matrix composites at large failure strain, without additional instrumentation. This method can be applied to determine the in-plane shear modulus  $G_{12}$ . According to Ref. [1], the shear stress corresponding to 5% strain (if destruction of the specimen does not occur earlier) is assumed as a failure criterion. Such a criterion is also recommended by the ASTM D 3518 standard [4].

According to Ref. [1], one load cycle is performed out at velocity given in a standard related to the tested material or 2 mm/min – in case of lack of such data. Investigation was finished at  $\gamma = 0.0500$ , if breaking of the specimen did not occur earlier. The shear modulus is defined in the interval of  $\gamma = 0.0010 \div 0.0050$ , and the shear strength corresponds to the shear stress at the failure of the specimen or at  $\gamma = 0.0500$ .

The method uses a tensile test of a  $[(\pm 45)_F]_{ns}$  laminate and can be applied in case of laminates with thermosetting matrices. The method is not suitable for investigation of laminates reinforced with thick fabrics. It is sensitive to number and distribution of the layers; the comparison is made using the same number of layers. The laminate of the  $\pm 45^\circ$  symmetrical configuration can be made of unidirectional or fabrics reinforced layers, provided that the number of fibers in the warp and the weft is equal to each other. During the tests, the specimen in the form of a rectangular beam with the fibers oriented at the angle of  $\pm 45^\circ$  to the specimen axis is subjected to an axial tension load. The fibers should be arranged symmetrically and in a sustainable manner with respect to the specimen axis.

It should be noted that the specimen material during the measurement is not in a state of pure shear, since in the  $\pm 45^\circ$  system there occur both shear and normal stresses. Up-to-date studies show that the normal stresses exhibit only negligible influence on the value of the shear modulus,  $G_{12}$ , and only slightly decrease the shear strength,  $R_{12}$  [12].

## 2.2. The tested material

The tests were carried out for the C/VE regular cross-ply laminate of  $[(0/90)_F]_{4S}$  configuration. The Style 430 fabric was produced by the ECC using carbon fibers Tenax-E HTA40, with fiber parameters provided in Tab. 1. The fabric is characterized by the following parameters: Style 430, a plain weave, 0.42 mm thickness, 1000 mm width, 300 g/m<sup>2</sup> substance, 400/400 warp/weft, 3.7/3.7 yarn/cm. The vinylester resin VE 11-M – applied for laminates with increased requirements for chemical resistance and incombustibility – was used to manufacture the considered composite. The VE 11-M resin parameters are given in Tab. 2.

Prefabricated laminate plates used in the investigations were produced by ROMA private enterprise; they were made using the vacuum molding method and the technological parameters developed by ROMA taking into account the VE 11-M datasheet.

*Tab. 1. Properties of the fiber (manufacturer data)*

L.P.	Property	Unit	Value
1	Sizing	-	E13 (Epoxy)
2	The content of the roving preparation	%	1.3
3	Number of filaments	-	6K (6000)
4	Nominal linear density	tex	400
5	Filament diameter	μm	7
6	Density	g/cm <sup>3</sup>	1.76
7	Tensile strength	MPa	3950
8	Young's modulus at tension	GPa	238
9	Elongation at break	-	0.017

*Tab. 2. Properties of the resin matrix (manufacturer data)*

L.P.	Property	Unit	Value	Standard
1	Tensile strength	MPa	80	ISO 527-2
2	Young's modulus at tension	GPa	3.5	ISO 527-2
3	Elongation at break	%	3.5	ISO 527-2
4	Flexural strength	MPa	120	ISO 178
5	Young's modulus at bending	GPa	4	ISO 178
6	Temperature of deflection under load	°C	85	ISO 75-2
7	Barcol hardness	°B	40	ASTMD 2583-95

### 2.3. Preparation of specimens

Plates of 550×550 mm dimensions, made using the vacuum molding method, were cut into small  $[(\pm 45)_F]_{ns}$  plates of length equal to the test specimen and of width appropriate for preparation of the required number of specimens. Additional cover plates were prepared in a similar manner. The cover plates were glued to the plate using a thin layer of adhesive (E-53 epoxy resin), according to [1]. The glued surfaces were degreased and sandpapered. The combined parts were tightened until the resin has been polymerized (12 h). Plates, together with the cover plates, constituted the initial material for cutting the specimens.

Rectangular prism shaped specimens were cut using a sawing machine with a diamond disk. The mechanically machined surfaces and edges of final specimens were free of imperfections visible through a magnifying glass with a low magnification.

Further processing was performed by milling. For roughing, made on the numerically controlled CNC BFN 7050SERVO AUTO milling machine, the speed 10000 rpm and feed rate 898 mm/min were used, and for the final treatment – 15000 rpm and 1224 mm/min, respectively. After the final treatment, the shape and dimensions of the specimens were compatible with standard [1] (Fig. 1, 2).

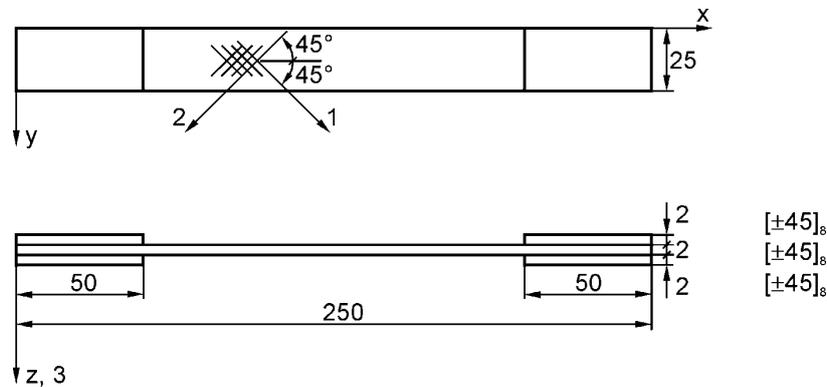


Fig. 1. A shape and geometry of the specimen, according to Ref [1]

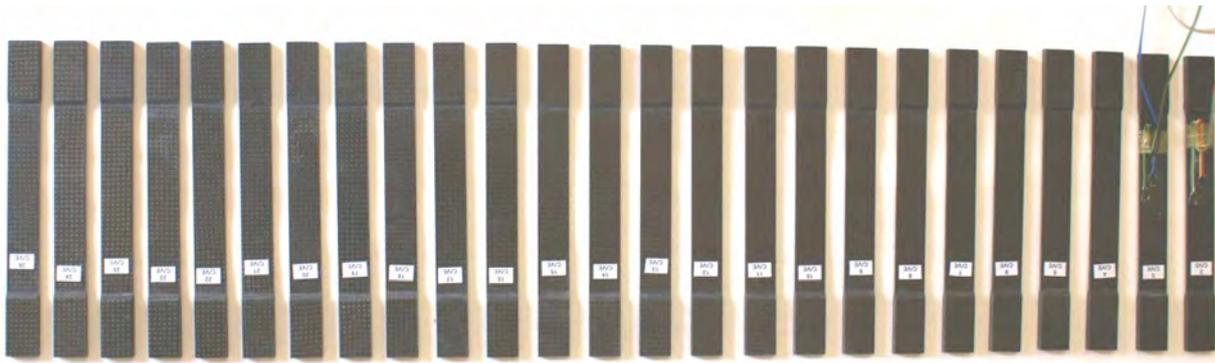


Fig. 2. Specimens for the shear test

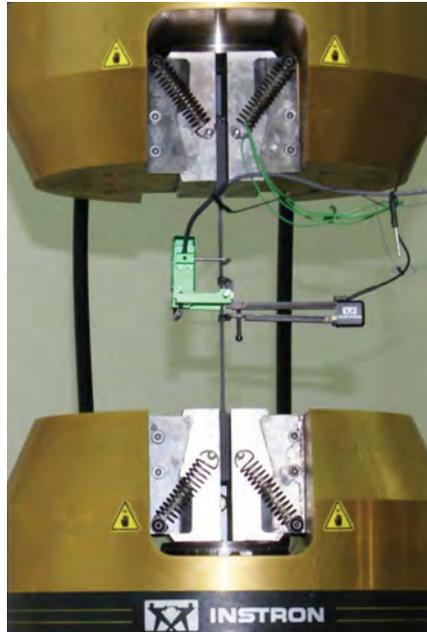
According to Ref. [1], the formed shapes were conditioned for more than 88 hours at temperature of  $23 \pm 1^\circ\text{C}$  and relative humidity of  $50 \pm 10\%$ . The tests were performed at the same conditions.

### 2.4. Instrumentation, measuring of the specimens, performing the tests

A width of the specimens was measured using a calliper of 0.1 mm accuracy, a thickness measured of 0.01 mm accuracy using a micrometer with the 2 mm cylinder foot. The measurements

were executed in accordance with Ref. [1]. Afterwards, the arithmetic mean of three measurement results was calculated. Longer edges of individual specimens were parallel with tolerance of 0.2 mm, therefore, respective standard requirements were fulfilled.

The investigations were executed on the INSTRON 8802 universal testing machine with hydraulic wedge grips (Fig. 3).

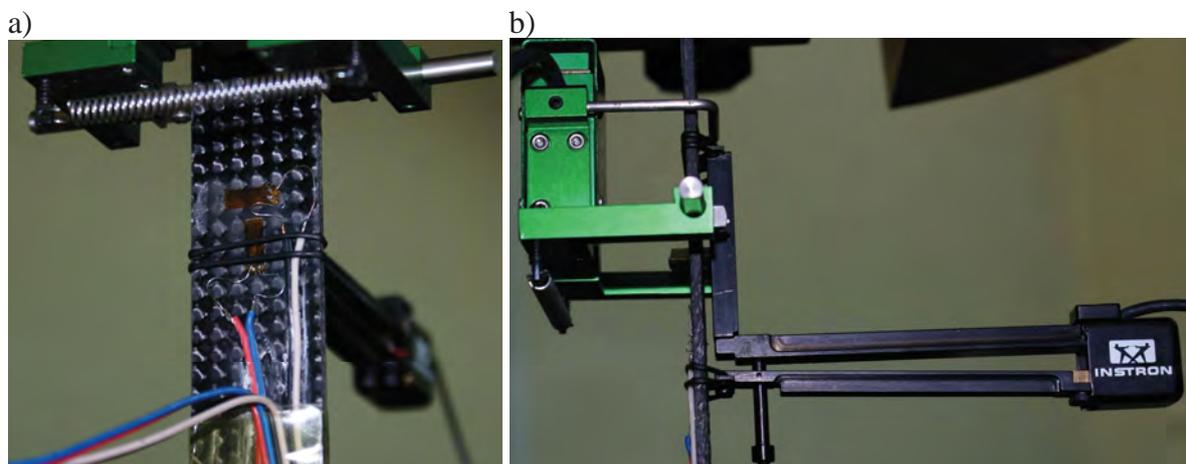


*Fig. 3. View of a specimen mounted in the hydraulic wedge grips*

Four different crosshead rates were used during the tests: 1, 2, 5 and 10 mm/min. The circuit of the load measurement showed the total spreading load transferred by the specimen and it did not show the inertial deceleration at every applied crosshead rate.

Using the FastTrack software, the measured values of displacement, force and deformation in real time were automatically recorded and saved on a hard disk with sampling frequencies of 5, 10, 25 and 50 Hz, respectively.

The measurement of deformation was performed in two directions, i.e., parallel and perpendicular to the axis of the specimen. Vishay strain gauges of the  $a \approx 6$  mm measuring base,  $120 \Omega$  resistance, sensitivity constant  $k = 2.08$ , and two extensometers – axial, INSTRON 2620-604, and transverse, INSTRON W-E404-E, – were used, both of the 25 mm measuring base (Fig. 4).



*Fig. 4. A specimen with installed sensors: a) strain gauges b) extensometers*

## 2.5. Processing of the measurement results [1]

The shear stress was calculated using the formulas resulting from rotation about the angle of 45° in uniaxial tension:

$$\tau_{12}(F) = 0,5\sigma, \quad \sigma = \frac{F}{bh}, \quad (1)$$

where:

$\tau_{12}$  - the current in-plane shear stress [MPa],

$F$  - the current load [N],

$b$  - width of the specimen [mm],

$h$  - thickness of the specimen [mm].

The in-plane shear strength is equal to

$$R_{12} = \tau_{12}(F_m), \quad (2)$$

where:

$F_m$  - load at break of the specimen or load corresponding to the deformation  $\gamma_{12} = 0.0500$ , if the test was completed before the specimen's failure [N],

$R_{12}$  - the in-plane shear strength (maximum shear stress before the moment of completion of the test or at the deformation  $\gamma_{12} = 0.0500$ ) [MPa].

The shear strain is determined from the formula resulting from the deformation in uniaxial tension and rotation about the angle of 45° in the layer plane, i.e.,

$$\gamma_{12} = \varepsilon_x - \varepsilon_y, \quad (3)$$

where:

$\gamma_{12}$  - shear strain (total deformation related to the directions parallel and perpendicular to the axis of the specimen),

$\varepsilon_x, \varepsilon_y$  - normal strains in the directions parallel and perpendicular to the axis of the specimen ( $\varepsilon_x > 0$ ,  $\varepsilon_y < 0$ ).

The shear modulus is calculated from the initial quasi-linear shear stress – shear strain relation:

$$G_{12} = \frac{\tau_{12}'' - \tau_{12}'}{\gamma_{12}'' - \gamma_{12}'}, \quad (4)$$

where:

$G_{12}$  - shear modulus [GPa],

$\tau_{12}'$  - shear stress corresponding to the strain of  $\gamma_{12}' = 0.0010$ ,

$\tau_{12}''$  - shear stress corresponding to the strain of  $\gamma_{12}'' = 0.0050$ ,

The shear stress – shear strain relation was determined for each specimen.

## 3. Test results and their analysis

### 3. 1. Evaluation of the usefulness of sensors used for strain measurements

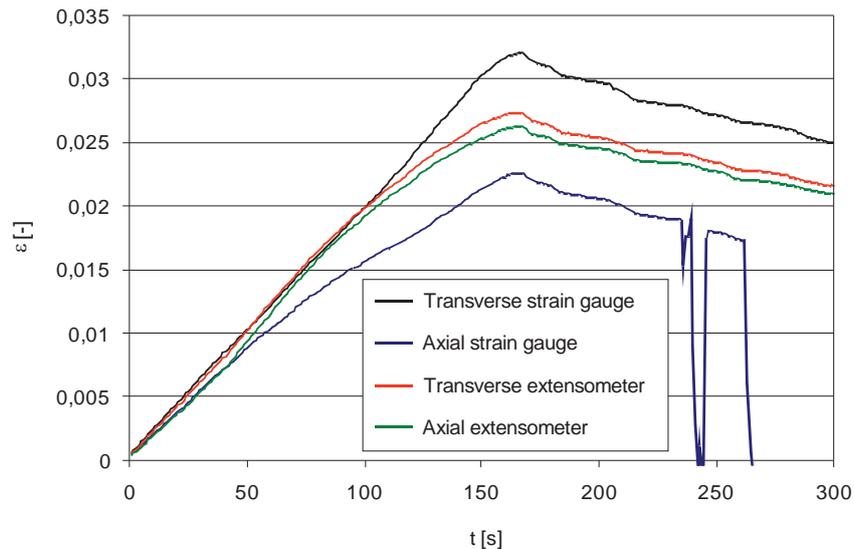
A comparative study was carried out in order to evaluate the usefulness of sensors for the strain measurement. Extensometers and strain gauges were installed on the same specimen (Fig. 4). Figure 5 shows the results of the measurement using strain gauges and extensometers versus time, at the rate of crosshead  $v = 1$  mm/min.

Significant differences in the strain recorded by the axial and transverse strain gauges and similar values of the strain recorded by the axial and transverse extensometers can be observed. This is caused by the stress distribution in the specimen. Strain gauges measured the strain in a small area close to the axis of the specimen, whereas extensometers measured the strain of the region covering the entire width of the specimen (gauges' base  $\approx 6$  mm, extensometers'

base  $\approx 25$  mm). The strain  $\gamma$  is the sum of the absolute values of strains  $\varepsilon_x$ ,  $\varepsilon_y$  and the strain  $\gamma$  for both types of sensors, calculated in this way, will be similar. The disadvantage of strain gauges is found in a smaller range of measured strain and in lack of reusability. The advantage of extensometers is averaging of shear strains in a large area of the specimen, ease and simplicity of installation.

The test (Fig. 5) ends at time  $t \approx 140$  s which corresponds to strain value of  $\gamma = 0.0500$ . Values recorded by a transverse strain gauge at the moments of  $t \approx 240$  and  $270$  s, are caused by short-circuiting.

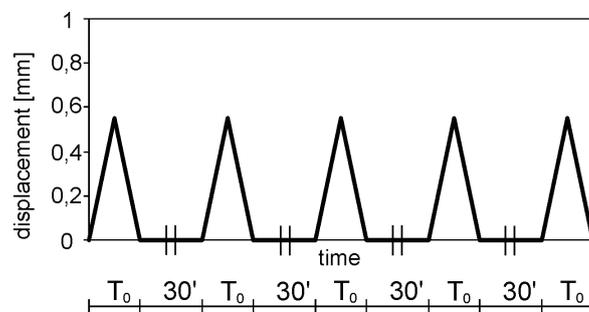
Taking into account the above preliminary analysis, only extensometers were used in further experiments.



*Fig. 5. Strains versus time recorded by strain gauges and extensometers for the selected specimen*

### 3.2. Results of the measurements and their analysis

In order to determine influences of a rate of the testing machine crosshead and of a number of stress cycles on the static relationship  $\tau$ - $\gamma$ , the loading program presented in Fig. 6 was assumed. Each specimen was subjected to five cycles of kinematic loading of a triangular shape, at intervals of 30' that protect finishing the reverse creep after each cycle. Each cycle contains a linear increase of the vertical displacement of the traverse up to 0.55 mm ( $\gamma = 0.0050$ - $0.0060$ ) and a linear decrease to zero.



*Fig. 6. The loading program for testing selected factors in the shear test*

The kinematic loading was executed at constant speed  $v = 1, 2, 5$  or  $10$  mm/min of the traverse. In this way, four five-cycle  $\tau$ - $\gamma$  diagrams for subsequent specimens were obtained, as shown in Figs. 7-10.

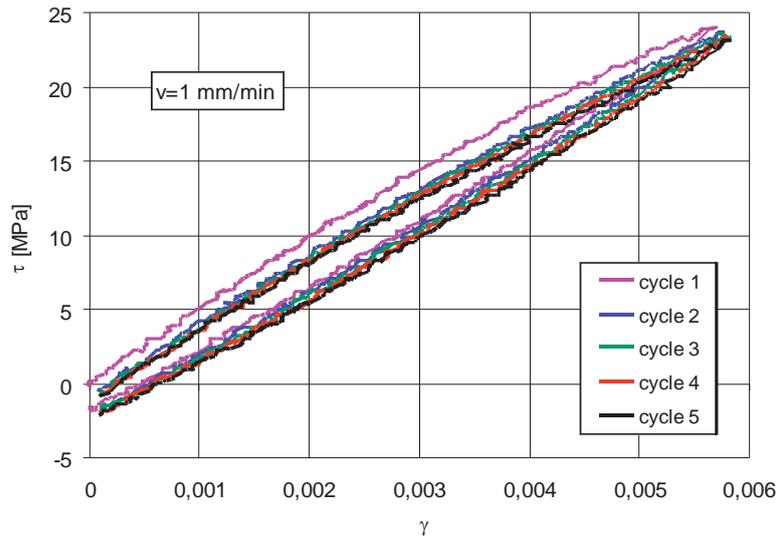


Fig. 7. The  $\tau$ - $\gamma$  diagram for  $v = 1$  mm/min (specimen No. 8)

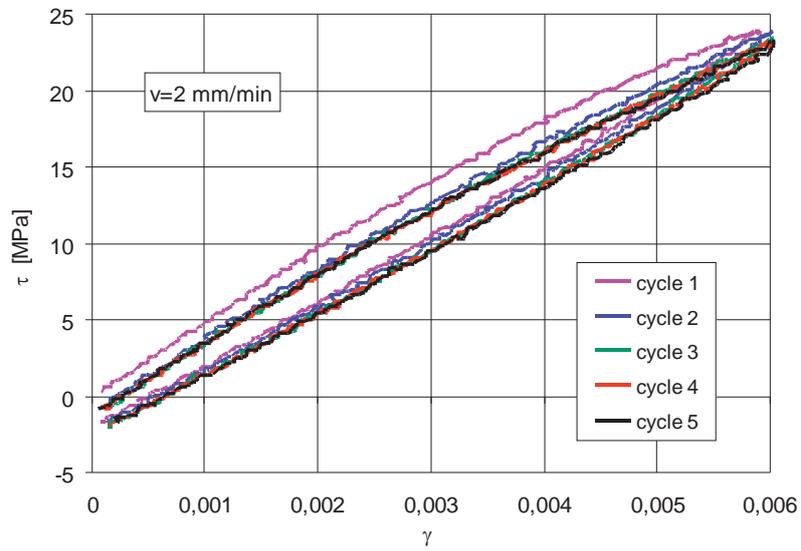


Fig. 8. The  $\tau$ - $\gamma$  diagram for  $v = 2$  mm/min (specimen No. 9)

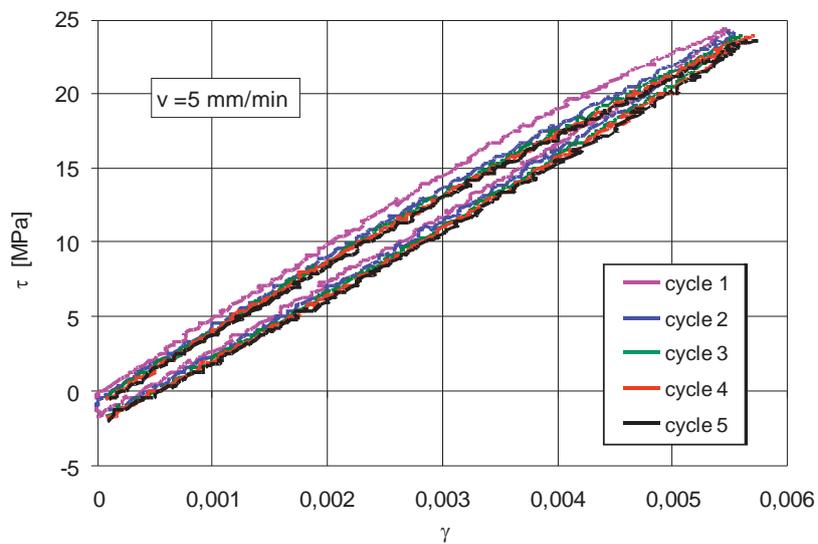


Fig. 9. The  $\tau$ - $\gamma$  diagram for  $v = 5$  mm/min (specimen No. 10)

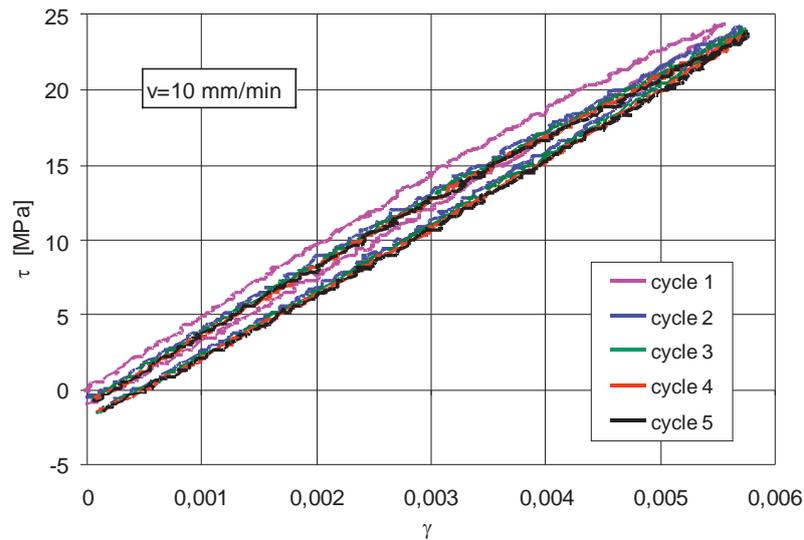


Fig. 10. The  $\tau$ - $\gamma$  diagram for  $v = 10$  mm/min (specimen No. 11)

Analysis of the  $\tau$ - $\gamma$  hysteresis loops presented in Figs. 7-10 leads to the following conclusions:

- 1) The executed strain interval corresponds to a linear Hooke's law. Small curvatures of plots in each cycle and a hysteresis loop indicate the occurrence of viscoelastic strains non-negligible for the tested crosshead rates and the stress range. Viscoelastic strains in the loading test are typical for laminates [14]. Evidence of this phenomenon is observed in the graph of the load versus time in the first cycle at  $v = 1$  mm/min (specimen No. 8), shown in Fig. 11 and in the graph of the  $\tau$ - $\gamma$  diagram shown in Fig. 12 for the strain range of  $\gamma = 0.0010 \div 0.0050$  proposed by ISO 14129 [1] in order to determine the shear module.
- 2) Along with increase of the crosshead rate a width of the hysteresis loop decreases. It corresponds to decrease of rheological effects.
- 3) After the first load cycle the permanent strain of  $\sim 0.0002$  value occurs. This strain corresponds to redistribution of the residual stresses in the laminate caused by the manufacture technology. During the stress increase in the first load cycle, there appear microcracks in the laminate, distributed quasi-uniformly in the whole volume of the specimen.
- 4) In the second and subsequent load cycles, the permanent strain increase is negligible. Therefore, in the 2<sup>nd</sup>, 3<sup>rd</sup>, 4<sup>th</sup>, 5<sup>th</sup> load cycles, further degradation of the laminate microstructure does not occur.
- 5) In the interval  $\gamma = 0.0010 \div 0.0020$ , the  $\tau$ - $\gamma$  relationship is quasi-linear for the tested traverse velocities, and thus the viscoelastic strains in this interval can be neglected (Fig. 13).

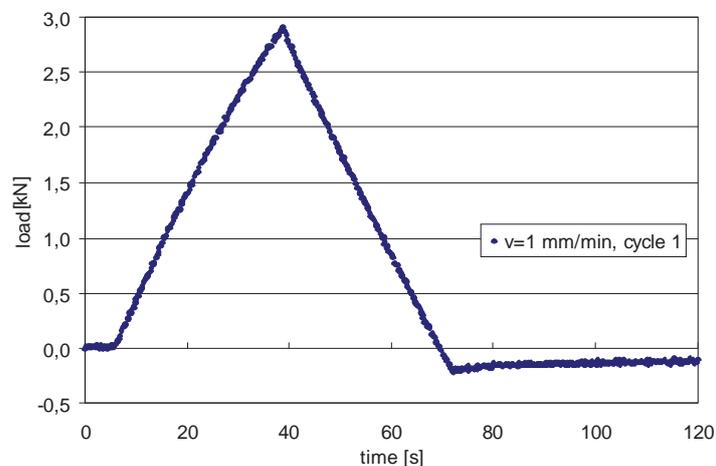


Fig. 11. The axial tensioning force versus time for  $v = 1$  mm/min in cycle 1 (specimen No. 8)

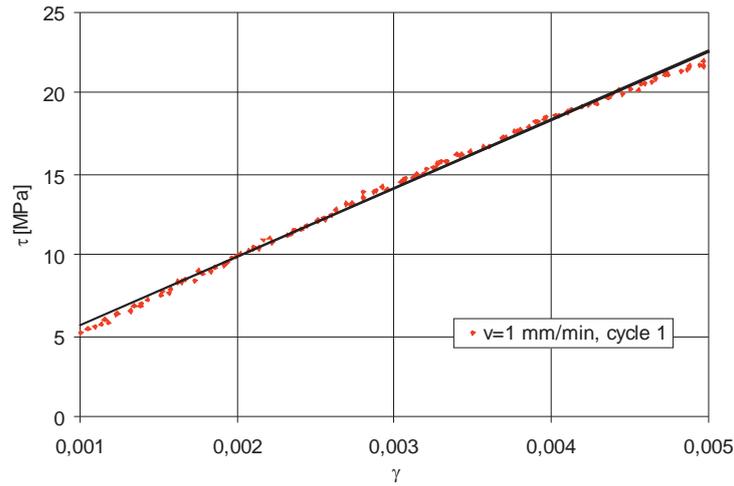


Fig. 12. The  $\tau$ - $\gamma$  diagram for  $v = 1$  mm/min in a cycle load its linear regression for  $\gamma = 0.0010 \div 0.0050$  (specimen 8)

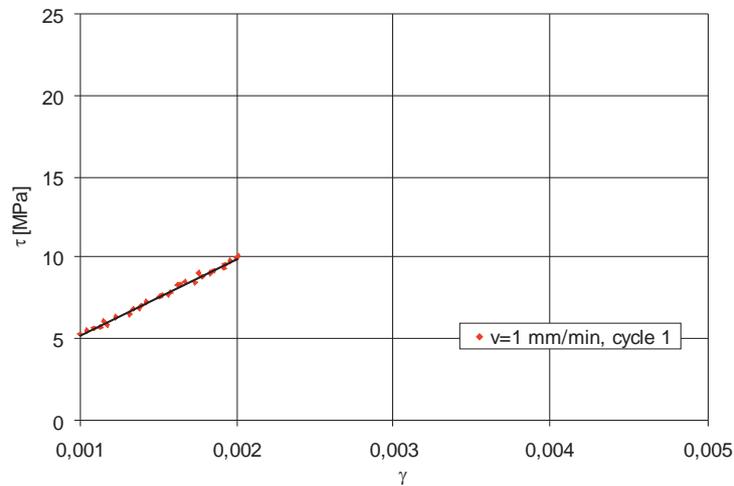


Fig. 13. The  $\tau$ - $\gamma$  diagram for  $v = 1$  mm/min in a cycle load its linear regression for  $\gamma = 0.0010 \div 0.0020$  (specimen 8)

The values of the shear modulus,  $G_{12}$ , for subsequent cycles of kinematic loading and applied load rates, calculated on the basis of the quasi-linear strain interval  $\gamma = 0.0010 \div 0.0020$ , are presented in Tab. 3 and Fig. 14.

Tab. 3. The shear modulus calculated for the strain interval  $\gamma = 0,0010 \div 0,0020$

Specimen No.	v [mm/min]	$G_{12}$ [GPa]				
		cycle 1	cycle 2	cycle 3	cycle 4	cycle 5
8	1	4.75	4.60	4.62	4.65	4.48
9	2	4.76	4.47	4.48	4.53	4.40
10	5	4.81	4.74	4.75	4.61	4.49
11	10	4.84	4.58	4.53	4.51	4.43

The values of the shear modulus,  $G_{12}$ , at the 2<sup>nd</sup>, 3<sup>rd</sup> and 4<sup>th</sup> load cycles can be considered as independent of the velocity of the crosshead rate and of a number of the loading cycles. In the first cycle, the shear modulus is greater by 6.4% (an average value) when compared to the second cycle results.

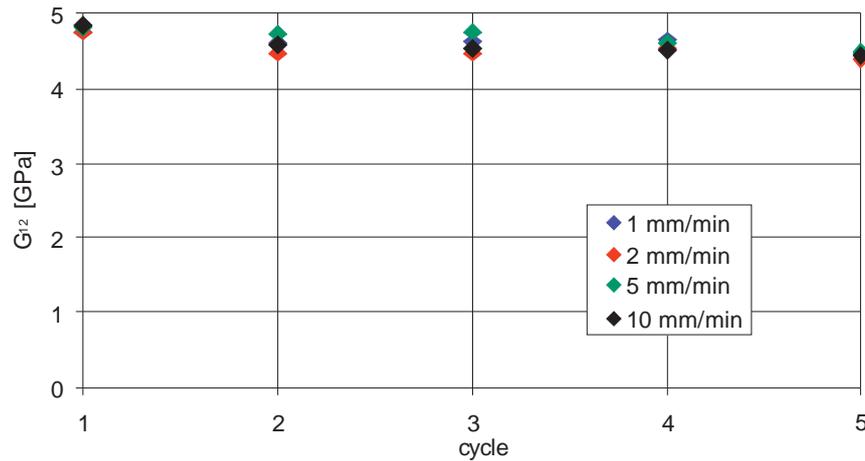


Fig. 14. The dependence of the shear modulus on a number of the loading cycles and on the crosshead rate

#### 4. Final conclusions

Taking into account notation in the PN-EN ISO 14129 standard [1] and detailed conclusions resulting from the experiments, the authors propose the following modified procedure concerning determination of selected mechanical properties of orthotropic laminates in the in-plane shear test:

- 1) Execution of the initial loading cycle of the triangular shape (linear increase of the crosshead displacement up to 0.55 mm and a linear decrease to zero), at the crosshead rate of  $v = 2$  mm/min) in order to redistribute the residual (technological) stresses in the specimen.
  - 2) A 30' break after the initial loading cycle, in order to perform the reverse creep of the specimen.
  - 3) Execution of the main test, i.e., linear increase of the crosshead displacement at the velocity of  $v = 2$  mm/min until the break of the specimen appears or the limited strain  $\gamma = 0.0500$  is reached. Determination of the  $\tau$ - $\gamma$  diagram.
  - 4) Determination of the shear modulus  $G_{12}$  based on the  $\gamma = 0.0010 \div 0.0020$  interval, using the linear regression due to measurement fluctuations.
  - 5) Determination of the shear strength  $R_{12}$  equal to the maximum value in the  $\tau$ - $\gamma$  diagram.
- Other requirements formulated in Ref. [1], related to the specimen's preparation and conditioning, the experiment performing and processing of the measurement results, are fully accepted and applied in this study.

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