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VERIFICATION OF THE STANDARD PROCEDURE OF THE MEASUREMENTS IN THE IN-PLANE SHEAR TEST OF A CROSS-PLY VINYLESTER-CARBON LAMINATE

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 plain weave carbon fabric of parameters: Style 430, Carbon 6K, substance 300 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 the ROMA private enterprise, Grabowiec, Poland, using the vacuum molding technology and the technological parameters developed by ROMA taking into account the VE 11-M material specification.

The PN-EN ISO 14129:2000 standard and closely related standards were taken into consideration in experimental studies on the static in-plane shear response by a tensile test of a $[(\pm45)_F]_{nS}$ 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. This procedure concerns the in-plane shear test and contains the following steps:

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 τ-γ diagram.**
4. **Determination of the in-plane 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 in-plane shear strength, R_{12}, equal to the maximum value in the τ-γ diagram.**

Keywords: **vinylester-carbon laminate, regular cross-ply laminate, in-plane shear test, in-plane shear modulus, in-plane shear strength, verification of standard procedures, statistical analysis**

WERYFIKACJA PROCEDURY NORMOWEJ POMIARÓW W PRÓBIE ŚCINANIA W PŁASZCZYŹNIE LAMINATU KRZYŻOWEGO WINYLOESTROWO-WĘGLOWEGO

Przedmiotem badań eksperymentalnych jest regularny laminat krzyżowy o konfiguracji $[0/90_F]_{4S}$. Warstwę laminatu stanowi żywica winyloestrowa VE 11-M (producent Zakłady Chemiczne „Organika-Sarzyna" S.A.) wzmocniona tkaniną węglową z przeplotem prostym (producent C. Cramer GmbH & Co. KG Division ECC) o parametrach: Style 430, włókno Carbon 6K, gramatura 300 g/m^2, osnowa/wątek 400/400 tex, 3,7/3,7 pasm/cm. Laminat został wytworzony przez przedsiębiorstwo ROMA Sp. z o.o. w Grabowcu z zastosowaniem technologii prasowania próżniowego i przyjętych parametrów technologicznych opracowanych przez ROMA z uwzględnieniem karty żywicy VE 11-M Zakładów Chemicznych w Sarzynie.

Przeanalizowano normę PN-EN ISO 14129:2000 oraz normy związane w zakresie badania ścinania statycznego w płaszczyźnie laminatu ortotropowego za pomocą jednokierunkowego rozciągania laminatu $[(\pm45)_{tk}]_{4S}$. Opracowano program badań eksperymentalnych ukierunkowany na zbadanie wpływu prędkości ruchu trawersy maszyny wytrzymałościowej oraz liczby cykli naprężenia statycznego na wyniki pomiarów modułu ścinania w płaszczyźnie laminatu. Na podstawie przeprowadzonych badań zaproponowano modyfikację procedury normowej w celu wyznaczenia poprawnej wartości modułu ścinania oraz wytrzymałości na ścinanie w płaszczyźnie laminatu.

Słowa kluczowe: **laminat winyloestrowo-węglowy, regularny laminat krzyżowy, badania na ścinanie w płaszczyźnie laminatu, moduł ścinania w płaszczyźnie laminatu, wytrzymałość na ścinanie w płaszczyźnie laminatu, weryfikacja przepisów normowych, analiza statystyczna**

INTRODUCTION

Shear tests for polymer-matrix composite materials are the most difficult tests for determining the mechanical proprieties of such materials. There are several methods of shear tests for laminates. There exists compatibility of these methods in relation to accuracy of the measurement and the value of the in-plane shear modulus, whereas determining the in-plane shear strength is more problematical. The edge effects, connecting of materials, non-linear behavior of the matrix or the matrix-fibers connection, stress distribution (imperfections), occurrence of the normal stresses cause that the value of the in-plane shear strength determined by existing methods is disputable. Therefore, the value of shear strength in engineering applications should be checked for each structure [1].

Some of the test methods were proposed in order to determine the τ-γ relation in the laminate plane, the other ones are confined to a plane perpendicular to the laminate plane and some - in dependence of the way of cut of the specimen - allow to research both cases. Summing up, the in-plane shear modulus and/or the in-plane shear strength are determined on the basis of a shear test. It is also possible to determine the failure strain [1].

The study has the following objectives: 1) determination of the influence of selected factors, i.e., a rate of the testing machine crosshead and a number of static loading cycles, on the shear stress - shear strain relationship; 2) formulation of the modified standard procedure for determination of the correct value of the in-plane shear modulus and the in-plane shear strength.

DESCRIPTION OF THE EXPERIMENTAL STUDIES

The shear test method corresponds to the PN-EN ISO 14129:2000 standard [2] and allows to determine the shear stress, τ, and the shear strain, γ, 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. [2], 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 [3].

According to Ref. [2], 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. 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 ±45° 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 ±45° to the specimen axis is subjected to an axial tension load. Normal stress in the differential element oriented at the angle of 45° to the longitudinal axis of the specimen is carried by high strength fabric fibers. Hence, the composite effort is mainly determined by shear stress due to a much lower in-plane shear strength R_{12}. 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 Table 1. The fabric is characterized by the following parameters: Style 430, a plain weave, 0.42 mm thickness, 1000 mm width, 300 g/m^2 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 Table 2.

TABLE 1. **Properties of the fiber (manufacturer data)**
TABELA 1. **Właściwości włókna (dane producenta)**

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

TABLE 2. **Properties of the resin matrix (manufacturer data)**
TABELA 2. **Właściwości matrycy żywicowej (dane producenta)**

Property	Unit	Value
Tensile strength	MPa	80
Young's modulus at tension	GPa	3.5
Elongation at break	%	3.5
Flexural strength	MPa	120
Young's modulus at bending	GPa	4
Temperature of deflection under load	°C	85
Barcol hardness	°B	40

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. Plates with dimensions of 550×550 mm and $[(0/90)_F]_{4S}$ structure were cut into small $[(\pm45)_F]_{4S}$ 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 Ref. [2]. The glued surfaces were degreased and sanded. 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.

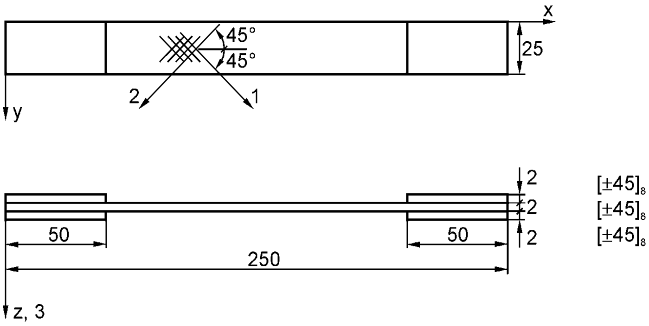

Fig. 1. A shape and geometry of the specimen, according to Ref. [2]

Rys. 1. Kształt i geometria próbki, zgodne z normą [2]

Specimen dimensions (Fig. 1) correspond to the PN-EN ISO 14129:2000 standard. Therefore, the Saint-Venant's principle is achieved in the allowed failure area. 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, carried out on the numerically controlled CNC BFN 7050SERVO AUTO milling machine with UFD28060 mill, the speed of 20000 rpm and feed rate of 1500 mm/min were used, whereas for the final treatment - 24000 rpm and 2700 mm/min.

The specimens were conditioned for more than 88 hours at temperature of 23 ±1°C and relative humidity of 50±10%. The tests were performed at the same conditions. The investigations were executed on the INSTRON 8802 universal testing machine with hydraulic wedge grips. Four different crosshead rates were used during the tests: 1, 2, 5 and 10 mm/min. 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.

Measurements of the strains 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 Ω 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.

PROCESSING OF THE MEASUREMENT RESULTS [2]

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

$$\tau_{12}(F) = 0.5\sigma, \quad \sigma = \frac{F}{bh} \tag{1}$$

where: τ_{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:

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

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 calculated using the formula resulting from the deformation in uniaxial tension and rotation about the angle of 45° in the layer plane, i.e.:

$$\gamma_{12} = \varepsilon_x - \varepsilon_y \tag{3}$$

where: γ_{12} - shear strain (total deformation related to the directions parallel and perpendicular to the axis of the specimen); ε_x, ε_y - normal strains in the directions parallel and perpendicular to the axis of the specimen (ε_x>0, ε_y<0).

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

$$G_{12} = \frac{\tau_{12}'' - \tau_{12}'}{\gamma_{12}'' - \gamma_{12}'} \tag{4}$$

where: G_{12} - shear modulus, GPa; τ_{12}' - shear stress corresponding to the strain of $\gamma_{12}' = 0.0010$; τ_{12}'' - shear stress corresponding to the strain of $\gamma_{12}'' = 0.0050$ (the standard procedure [2]) or to the strain of $\gamma_{12}'' = 0.0020$ (the modified standard procedure). The shear stress - shear strain relation was determined for each specimen.

THE EXPERIMENTAL RESULTS AND THEIR ANALYSIS

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. 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 specimen axis, whereas extensometers measured the strain of the region covering the entire width of the specimen (gauges' base ≈ 6 mm, extensometers' base ≈ 25 mm). The strain γ is the sum of the absolute values of strains ε_x, ε_y and the strain γ 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.

In order to determine influences of a rate of the testing machine crosshead and of a number of stress cycles on the static relationship τ-γ, a loading program presented in Figure 2 was assumed. Each specimen was subjected to five cycles of kinematic loading of a triangular shape. The cycles are of low amplitude and their duration was ranging from 6.6 to 66 s. The reverse creep time was established as 30 minutes that allows almost complete reduction of viscoelastic deformation after each cycle. Each cycle contains a linear increase of the vertical displacement of the traverse up to 0.55 mm (γ = 0.0050÷0.0060) and a linear decrease to zero.

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 τ-γ diagrams for subsequent specimens were obtained; the first of them is shown in Figure 3.

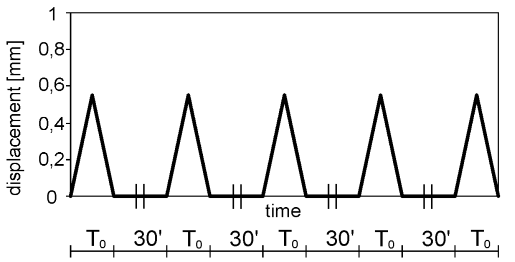

Fig. 2. The loading program for testing selected factors in the shear test

Rys. 2. Program obciążenia do testowania wybranych czynników w próbie ścinania

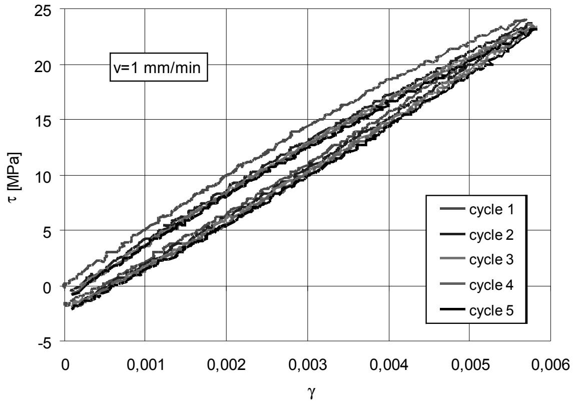

Fig. 3. The τ-γ diagram for v = 1 mm/min (specimen No. 8)

Rys. 3. Wykres τ-γ przy prędkości v = 1 mm/min (próbka nr 8)

Analysis of the τ-γ hysteresis loops 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.
2. Along with increase of the crosshead rate a width of the hysteresis loop decreases. It corresponds to obvious decrease of rheological effects.
3. After the first load cycle, the permanent strain of ~ 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, increase of the permanent strains is negligible. Therefore, in the 2nd , 3rd , 4th , and 5th load cycles, further degradation of the laminate microstructure does not occur.
5. In the interval γ = 0.0010÷0.0020, the τ-γ relationship is quasi-linear for the tested traverse velocities, and thus, the viscoelastic strains in this interval can be neglected (Fig. 4).

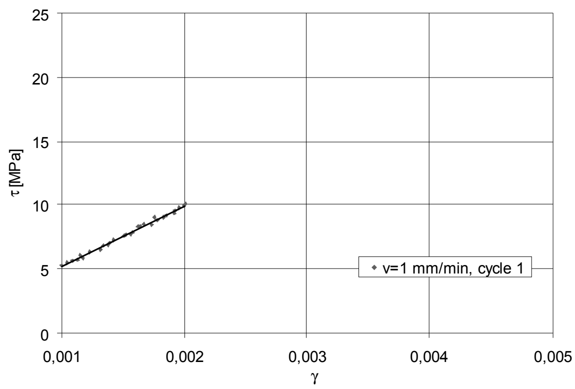

Fig. 4. The τ-γ diagram for v = 1 mm/min in 1st load cycle and its linear regression for γ = 0.0010÷0.0020 (specimen No. 8)

Rys. 4. Wykres τ-γ przy prędkości v = 1 mm/min w cyklu 1 oraz jego liniowa regresja dla γ = 0,0010÷0,0020 (próbka nr 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 γ = 0.0010÷0.0020, are presented in Table 3.

TABLE 3. **The shear modulus calculated for the strain interval γ = 0.0010÷0.0020**

TABELA 3. **Moduł ścinania obliczony z wykorzystaniem przedziału γ = 0,0010÷0,0020**

v	G_{12}, GPa				
mm/min	cycle 1	cycle 2	cycle 3	cycle 4	cycle 5
1	4.75	4.60	4.62	4.65	4.48
2	4.76	4.47	4.48	4.53	4.40
5	4.81	4.74	4.75	4.61	4.49
10	4.84	4.58	4.53	4.51	4.43

The values of the shear modulus, G_{12}, at the 2nd, 3rd and 4th 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.

FINAL CONCLUSIONS

Taking into account notation in the PN-EN ISO 14129:2000 standard [2] and conclusions resulting from the executed 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 τ-γ 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 τ-γ diagram.

Other requirements formulated in Ref. [2], 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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