



UNIVERSITY OF TRENTO

Department of Industrial Engineering Composite Materials Engineering

Professors : Pegoretti A., Mahmood H.
Sergio Maria Leso
MAT [REDACTED]

Abstract

Basalt fibers are natural fibers with good mechanical properties, the tensile modulus and strength are comparable with glass fibers considering them as a valid alternative to those one^[1]. The most common thermoset matrix used is the epoxy resin thanks to its high resistance to moisture absorption and to corrosive liquids^[2]. A general study of composite material was carried using different mechanical test and thermogravimetric test. The evaluation starts from single fibers tensile test, the laminates preparation in order to form the composite material with the study of fiber volume fraction and volume presence of voids. The matrix was analysed under differential scanning calorimetry to assess the presence of any residual curing of the thermoset matrix. Fracture and mechanical properties of the final composite laminates were analysed at the end. 0°/90° and ±45° laminates were analysed, tensile modulus and strength were evaluated and found higher than glass fibers composites and similar to carbon fibers composites.

Tensile testing of single fibers

Introduction

Composite materials are composed by fibers and matrix with different properties. The combination of the mechanical and fracture properties of the components affect the final mechanical properties of the composite material^[3]. The single fiber tensile test is useful to study the failure properties of single basalt fiber.

Materials and Methods

In order to study the mechanical and fracture properties of single fibers, a tensile test was carried on a woven fabric of basalt fibers BAS220. 1270.T from BASALTEX™.

The single fibers tensile testing was carried following ASTM C1557^[4] with a strain rate of 0.5 min⁻¹. Different gauge lengths were analysed in order to determine the scale and shape parameter of Weibull distribution (15 mm, 30 mm, 60 mm). To determine a correct value of tensile strength and Young's modulus the number of specimen was : 16 specimen of L₀=15

mm, 29 specimen of L₀=30 mm, 18 specimen of L₀=60 mm.

The scale and shape parameter of the Weibull distribution were analysed with three different methods.

Method 1 – Tensile test for single length fibers

The first method is based on a fracture test on a sample with specimens of the same length L₀, in this case L₀ = 30 mm. The number of specimen analysed under fracture test were 29. After evaluating the strength values of fracture (σ, in MPa), a particular index was assigned from 1 to N. N is the number of specimen studied. Function was assigned following the Eq.1 :

$$F(\sigma) = \frac{i-0.5}{N} \quad (1)$$

where F(σ) represents the probability of failure. In order to find the shape parameter m and the scale parameter σ₀ the Weibull distribution was linearized.

The slope of linearized Weibull distribution will give the shape parameter. The scale parameter was evaluated using Eq. 2 :

$$\sigma_0 = e^{\left(-\frac{\text{Slope}}{\text{Intercept}}\right)} \quad (2)$$

Method 2 -Tensile test for different lengths fibers

The second method was employed to find the scale and shape parameter related to different sample with various lengths, giving a more generalize analysis. The average strength of the Weibull distribution is given by :

$$\bar{\sigma} = \sigma_0 \left(\frac{L}{L_0}\right)^{-\frac{1}{m}} \Gamma\left(1 + \frac{1}{m}\right) \quad (3)$$

where Γ is the gamma function, L_0 is the reference length and m the shape parameter. The Eq.3 is linearized giving :

$$\ln \bar{\sigma} = -\frac{1}{m} \ln \left(\frac{L}{L_0}\right) + \ln \sigma_0 + \ln \Gamma\left(1 + \frac{1}{m}\right) \quad (4)$$

Using Eq.4 shape and scale parameters can be find.

Method 3

The third method is based on Pegoretti, Di Benedetto and Gurvich work^[6], a iterative procedure is employed for determination of shape and scale parameter of a Weibull distribution. The evaluation starts with an unknown value related to the shape parameter. After the iteration we have to verify that the $|m - m'| \leq 0.01$, where m' is the found shape parameter after the first iteration.

Young's modulus and system compliance

The evaluation of the Young's modulus and system compliance was carried following the ASTM C1557. The tensile test was carried for all the specimen with different lengths, the cross-head displacement is given by:

$$\Delta L = \Delta l + C_s F \quad (5)$$

where ΔL is the recorded cross-head displacement, in meters, F is the force to failure in N, Δl is the elongation of the specimen gage length, in meters and C_s is the system compliance in m/N.

The strain for a fiber is given by :

$$\varepsilon = \frac{\sigma}{E} = \frac{F}{EA} = \frac{\Delta l}{l_0} \quad (6)$$

Combining Eq.5 and Eq.6 :

$$\frac{\Delta L}{F} = \frac{\Delta l}{F} + C_s = \frac{l_0}{EA} + C_s \quad (7)$$

From Eq.6 the Young's modulus and the system compliance can be evaluated.

Experimental results

The probability of failure versus the stress at failure is represented in Fig.1. The fracture test was carried on 29 specimen with length of 30 mm.

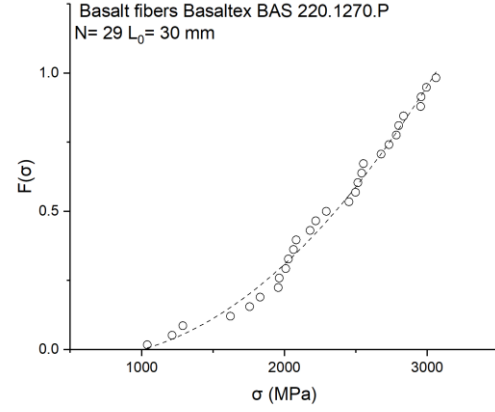


Fig. 1 – Probability to failure vs stress at failure. The shape is characteristic of a Weibull distribution.

The Weibull distribution was linearized:

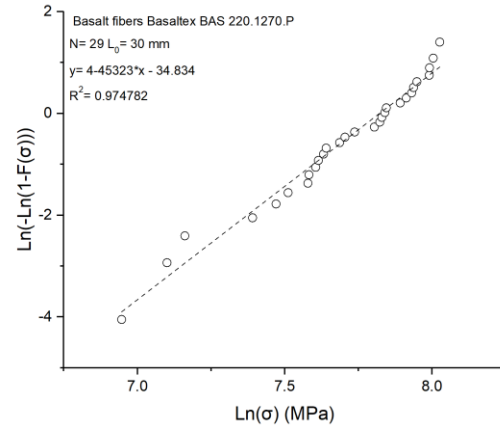


Fig. 2 – Linearized Weibull distribution.

From the slope in Fig.2 the shape parameter is obtained and the σ_0 (scale parameter) can be evaluated. The results are resumed in Table 1.

The second method is based on the evaluation of the average value of the stress at failure and it will give the shape and scale parameter for fibers with different lengths. From Eq. 4 the plot of $\ln(L/L_0)$ versus $\ln(\sigma)$ was used to evaluate the shape and scale parameter.

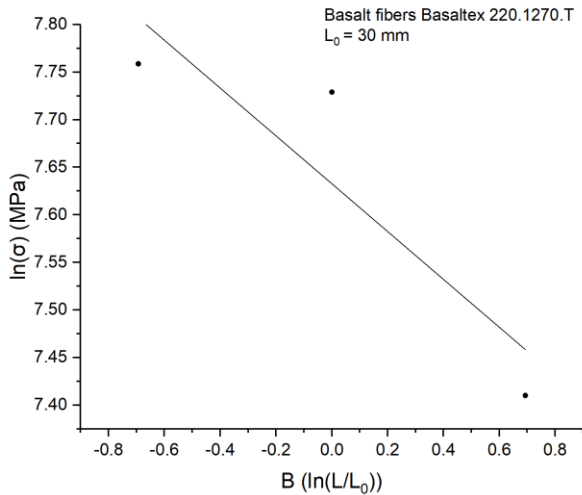


Fig. 3 – Second method applied, the data represents the average stress failure for 3 different lengths.

The results of method 2 and the iterative mode 3 analysis are resumed in Table 1.

	Shape parameter m	Scale parameter σ_0 [MPa]
Method 1	4.45323	2496.58
Method 2	3.9768	2278.4
Method 3	3.4173	2359.41

Table 1 – Shape parameter and scale parameter of three different methods to evaluate it.

The Young's modulus can be evaluated plotting the $\Delta L/F$ versus l_0/A . The inverse of the slope will give the elastic modulus. The intercept of the curve will give the system compliance.

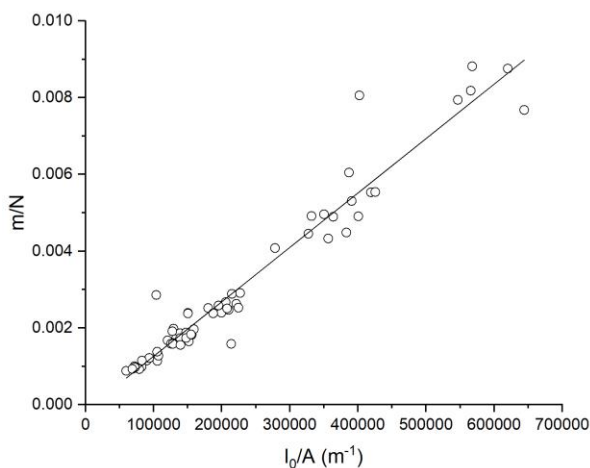


Fig. 4 – ASTM C1557 method applied to find the elastic modulus and the system compliance.

In Table 2 the Young's modulus and the system compliance are reported.

Young's modulus [GPa]	System compliance
70.52	-0.000156

Table 2 – Young's modulus and system compliance for basal fibers analysed.

Data analysis

The first method analysed has the limitation that analyse only a reference fiber length not taking into account the possibility of having different length. The second and the third method possess a more general study of the shape and scale parameters of different length of the Basalt fibers.

High value of the shape parameter is a good results meaning that the failure of basalt fiber is not high dispersive and it's easier to predict the failure life.

The scale parameter is similar for all the techniques, the scale parameters represents the shift of the Weibull distribution respect the failure stress, higher scale parameter higher resistance to failure. The elastic modulus found is similar to average values of single basalt fiber^[5].

Conclusion

Single test fibers were analysed in order to evaluate the failure behavior and the elastic modulus. The shape parameter for a population of basal fibers with different legnthns (15 mm, 30 mm and 60 mm) was in a range from 3.42 to 3.97. The scale parameter is similar for method 2 and method 3.

The elastic modulus found is equal to 70.52 GPa with a system compliance of -0.000156.

Reference

- [1]V. Fiore, T. Scalici, G. Di Bella, A. Valenza,A review on basalt fibre and its composites,Composites Part B: Engineering,Volume 74, 2015,Pages 74-94,ISSN 1359-8368,<https://doi.org/10.1016/j.compositesb.2014.12.034>.
- [2]Jingjing He, Junping Shi, Xiaoshan Cao, Yifeng Hu, "Tensile Mechanical Properties and Failure Modes of a Basalt Fiber/Epoxy Resin Composite Material", Advances in Civil Engineering, vol. 2018, Article ID 7914727, 10 pages, 2018.
<https://doi.org/10.1155/2018/7914727>
- [3]Tri-DungNgo, Introduction to Composite Materials, Composite and Nanocomposite Materials - From Knowledge to Industrial Applications, 2020, IntechOpen, DOI: 10.5772/intechopen.91285.
- [4]ASTM C1557-20, Standard Test Method for Tensile Strength and Young's Modulus of Fibers, ASTM International, West Conshohocken, PA, 2020, www.astm.org
- [5]Jongsung Sim, Cheolwoo Park, Do Young Moon,Characteristics of basalt fiber as a strengthening material for concrete structures, Composites Part B: Engineering, Volume 36, Issues 6–7, 2005, Pages 504-512, ISSN 1359-8368,
<https://doi.org/10.1016/j.compositesb.2005.02.002>.
- [6] Gurvich, M.R., Dibenedetto, A.T. & Pegoretti, A. Evaluation of the statistical parameters of a Weibull distribution. Journal of Materials Science 32, 3711–3716 (1997).
<https://doi.org/10.1023/A:1018603118573>

Introduction

Basalt fibers can be embedded inside a matrix with different method, a Vacuum Assisted Resin Transfer Molding was employed to create the composite material. Properties of the resultant material were analysed, like fiber volume fraction and voids content.

Materials and Methods

The basal fiber-epoxy matrix composite material was created using Vacuum Assisted Resin Transfer Molding (VARTM). The basalt fabric produced by Basaltex 220.1270.P was 4 layer balanced 0°/90° fiber orientation. The areal weight of basalt fibers was 220 g/m² and the density was equal to 2.634 ± 0.005 g/cm³. The epoxy resin employed was produced by Elantas composed by the base epoxy (EC157.1) and hardener (W342) with mix ratio of 100/30, the provided density from the producer was 1.147 ± 0.007 g/cm³. After using VARTM the composite material was cured at 100 °C for 8 hours in hot press under compressive load of 5 tons. The resulting composite material was analysed by a displacement method in ethanol (Archimedes method).

In order to evaluate the fiber volume fraction and the voids content of the produced composite laminate the starting basalt fibers and the final composite material were weighted using a Gibertini E42 balance with an error of ±0.01 g.

The fiber weight fraction was analysed using :

$$W_f = \frac{w_{basalt\ fabric}}{w_{composite}} \quad (1)$$

where W_f is the fiber weight fraction, $w_{basalt\ fabric}$ is the weight of the basalt fabric and $w_{composite}$ is the weight of the composite. The matrix weight fraction can be calculated substituting to the weight of the basalt fabric the weight of epoxy resin matrix.

The composite density can be evaluated using the following equation :

$$\rho_{composite} = \frac{1}{\left(\frac{W_f}{\rho_{basalt\ fiber}}\right) + \left(\frac{W_{matrix}}{\rho_{epoxy\ resin}}\right)} \quad (2)$$

The fiber volume fraction can be calculated as :

$$V_f = \frac{\rho_{composite}}{\rho_{basalt\ fiber}} W_f \quad (3)$$

The voids volume of the composite material is equal to :

$$V_v = \frac{\rho_{composite} - \rho_{experimental\ composite}}{\rho_{composite}} \quad (4)$$

Experimental Results

The weight of the basal fabric and of the composite material are resumed in Table 1.

Basalt fabric weight [g]	Composite weight [g]
33.06 ± 0.01	41.63 ± 0.01

Table 1 – Weight result for basal fabric and composite weight using Gibertini E42.

The experimental density find using displacement method in ethanol was equal to 2.061 ± 0.036 g/cm³.

Applying the Eq. 1,2,3,4 the results are resumed in Table 2.

W_f	0.7941	
W_m	0.2058	
$\rho_{composite}$	2.079 ± 0.036	g/cm ³
V_f	0.6268 ± 0.011	
V_v	0.0087	0.87 %

Table 2 – Weight fraction, volume fraction and density of produced composite material.

The volume of voids content is equal to 0.87% of the volume fraction of the composite material.

Data analysis

The difference value between the experimental density is slightly different from the theoretical one due to the presence of voids. The value of residual voids is acceptable. The value can be considered acceptable if under 1% of the whole volume fraction^[1].

Conclusion

The composite material was evaluated after VARTM, the laminate has a 62% of fiber volume fraction and a voids volume of 0.87%. The presence of voids lower than 1% of the total weight fraction is an indication of good quality material for aeronautical application. In other application 5% of voids content can be tolerated, the studied composite possess very low voids content.

Reference

[1]Mehdikhani M, Gorbatikh L, Verpoest I, Lomov SV. Voids in fiber-reinforced polymer composites: A review on their formation, characteristics, and effects on mechanical performance. Journal of Composite Materials. 2019;53(12):1579-1669. doi:10.1177/0021998318772152

Introduction

Thermoanalytical techniques can be used for evaluating material properties like melting temperature, glass transition temperature, cure process, thermal stability and crystallization kinetic^[1]. Differential Scanning Calorimetry (DSC) was carried on a sample of treated epoxy/basalt composites, treated epoxy resin and untreated epoxy resin.

Materials and Methods

DSC analysis was carried in order to study the glass transition temperature and curing effect of thermal treatment on composite sample and epoxy resin sample. In order to compare the results a DSC analysis on an untreated epoxy resin sample was carried. The composite sample was weighted using a Gibertini E42 (with error of 0.01 g).

The treatment on the composite and on the resin was a thermal treatment of 100 °C for 8 hours. The analysis used a Mettler Toledo DSC30 under a nitrogen flux of 100 mL/min, with an heating-cooling-heating cycle from 0 °C to 150 °C.

Experimental Results

In Figure 1 the DSC diagram are resumed. Fig.1 a) represent the DSC of untreated resin epoxy, an exothermic reaction is clearly visible in untreated sample, the epoxy resin is a thermoset polymer and the presence of big exothermic it can be the result of curing process in act.

Fig.1 b) represent the DSC of treated resin epoxy and in Fig.1 c) represents the DSC of treated composite material. The treated sample does not present exothermic peak in the first heating cycle.

The glass transition temperature was evaluated and the results are reported in Table 1.

	First Heating	Second Heating
Untreated epoxy	102.74 °C	79.26 °C
Treated epoxy	95.28 °C	100.98 °C
Treated composite	103.33 °C	101.16 °C

Table 1 – Glass transition temperature in the first heating and second heating of uncured epoxy, treated epoxy and treated composite.

Data Analysis

The presence of exothermic peak in the untreated epoxy resin sample is a signal related to the curing process. The matrix without any treatment with the increasing of the temperature starts its curing process that comports an increasing of the glass transition temperature. This particular exothermic peak is not present in the treated sample. The thermal treatment carried for 8 hours at 100 °C justify the absence of any

residual curing. The matrix is fully cured after the treatment.

The glass transition temperature is increased in the composite material respect the treated epoxy resin matrix thanks to the presence of the basalt fibers.

Conclusion

The absence of any residual curing process after thermal treatment means that the treatment was effective in order to cure the native epoxy resin. When we are building a composite material with a thermoset matrix is important to fully cure the material in order to give high mechanical properties^[2]. The glass transition temperature in case of epoxy-basalt composite is increased respect the treated epoxy resin .

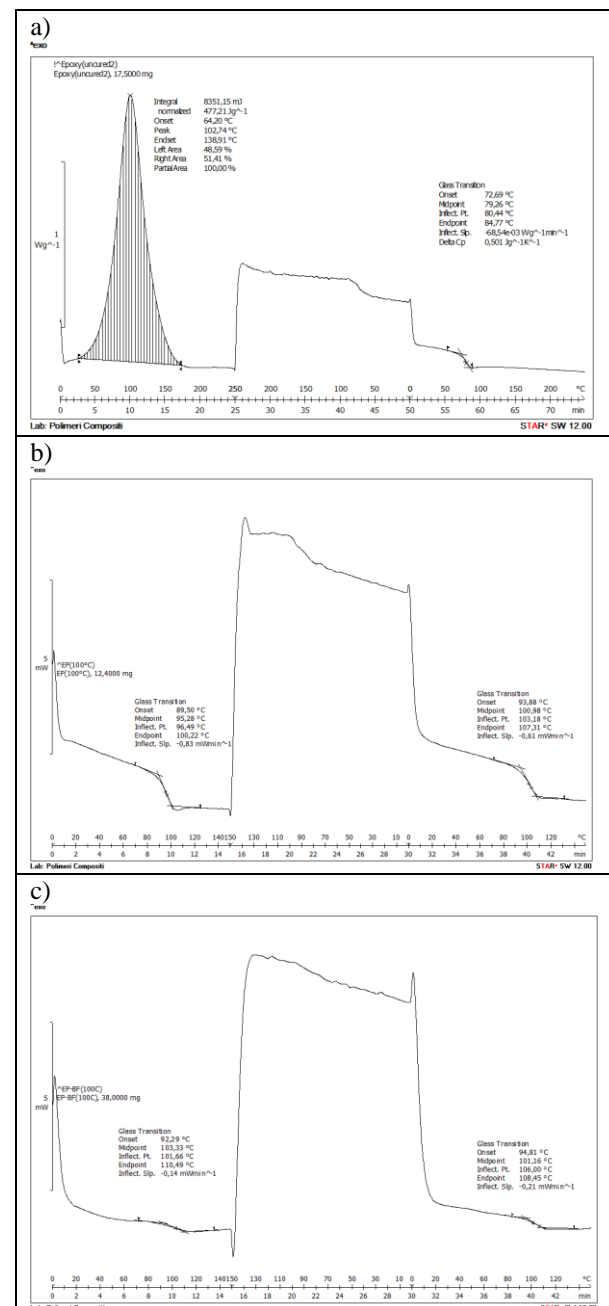


Fig. 5 – a) DSC of untreated epoxy resin, b) DSC of treated epoxy resin, c) DSC of composite material.

Reference

[1] Qiuju Zheng, Yanfei Zhang, Maziar Montazerian, Ozgur Gulbitten, John C. Mauro, Edgar D. Zanotto, and Yuanzheng Yue, Understanding Glass through Differential Scanning Calorimetry, Chemical Reviews 2019 119 (13), 7848-7939
DOI: 10.1021/acs.chemrev.8b00510

[2] Overview of material for Epoxy Cure Resin, MatWeb database,
http://www.matweb.com/search/datasheet_print.aspx?matguid=956da5edc80f4c62a72c15ca2b923494

Introduction

Tensile test on fibers reinforced polymer composite is used to determine in-plane tensile properties of material. A BF/ER (basalt fibers- epoxy resin) was tested in order to evaluate different mechanical properties, the major Poisson's ratio, the Tensile modulus, the Ultimate Tensile Strength (UTS) and the ultimate tensile strain.

Materials and Methods

Different configuration of composite material made by basalt fibers from Basaltex 220.1270.P and epoxy resin from Elantas (EC157.1, W342, mixing ratio 100:30) were test using a universal testing machine with load cell of 50 kN. The fiber-reinforced polymer composite were produced using VARTM, two different configurations were studied, 0°/90° and ±45° laminate orientation. In order to compare the properties a epoxy resin sample was analysed.

The specimens were cut from laminates according to ASTM D3039[1], different specimen were analysed : 6 specimen of epoxy resin only material, 4 specimen for composite material 0°/90° and 4 specimen composite material ±45°.

The Poisson's ratio was analysed using a bi-axial extensometer by Instron 2620-613. The gauge lengths for longitudinal and lateral extensometer were 12.5 mm and 25 mm respectively. The test was carried until a maximum deformation of 0.5% was present on the specimen, the cross-head speed was 2 mm/min. The Poisson's ratio was calculated using the following equation :

$$\nu = -\frac{\Delta\epsilon_t}{\Delta\epsilon_l} \quad (1)$$

where, ν is the Poisson's ratio, $\Delta\epsilon_t$ transversal strain respect the tensile test direction and $\Delta\epsilon_l$ longitudinal strain.

The tensile modulus was analysed using an extensometer applied on the face and in the centre of the specimen. Longitudinal extensometer Instron 2620-601 was employed with a gauge length of 25 mm. Maximum deformation of 0.5% was applied and the calculation of the modulus followed the ASTM D3039. The cross-head speed was 2 mm/min. The tensile modulus :

$$E = \frac{\Delta\sigma}{\Delta\epsilon} \quad (2)$$

where, E (in GPa) is the tensile modulus of elasticity, $\Delta\sigma$ (in MPa) the applied stress and $\Delta\epsilon$ the longitudinal strain.

The UTS and the ultima tensile strain were calculated applying a tensile test until fracture using a universal testing machine with cross-head speed of 10 mm/min.

The ultima tensile strain was calculated using :

$$\text{Ultima tensile strain} = \frac{\text{Maximum estension [mm]}}{\text{Total lenght of specimen[mm]}} \quad (3)$$

Lastly the shear stress τ_{LT} versus the shear strain γ_{LT} was analysed for the laminates ±45° following the ASTM D3518[2]. The shear stress was calculated using :

$$\tau_{LT} = \frac{F_i}{2A} \quad (4)$$

where, P_i is the force at i-th data point (in N) and A is the cross-sectional are (in mm²). The shear strain is equal to :

$$\gamma_{LT} = \epsilon_L - \epsilon_T \quad (5)$$

where, ϵ_L is the strain in longitudinal direction and ϵ_T is the transversal strain.

Experimental Results

The Poisson's results, the tensile modulus, the UTS and ultimate tensile strain are resumed in Table 1.

	Epoxy resin	Laminate ±45°	Laminate 0°/90°
Poisson's ratio ν		0.54	0.19
Tensile modulus E [GPa]	4.64	11.3	23.9
UTS [MPa]	51.1	99.4	372
Ultima tensile strain	7.5 %	32.1 %	2.8 %

Table 1 – Poisson's ratio, Tensile modulus, UTS and ultimate tensile strain results.

The stress strain curve of epoxy resin, ±45° laminates and 0°/90° are reported in Fig.1 in a), b) ,c) respectively. The shear stress versus the shear strain are reported in Fig.2. The shear modulus is equal to 11609 MPa.

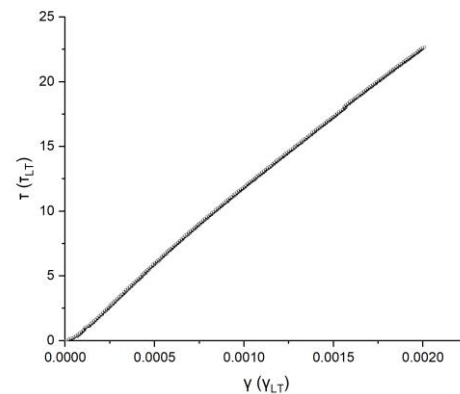


Fig. 2 – Shear stress versus shear strain for ±45 °.

Data analysis

The results resumed in Table 1 are similar to Carbon fibers composites and higher than glass fiber composites, meaning the good properties of BF/ER composites^[3]. The Poisson's ratio is very dependent on the orientation of the ply in the laminates.

The stress and strain curve for the cross-ply laminates in Fig.1 a) shows the presence of 90° ply failure at around 250 MPa, the effect of ply failure is shown by the change in the slope in the stress and strain curve.

The stress and strain curve for $\pm 45^\circ$ laminates shows a very complex shape, with a ply failure at very low strain.

Conclusion

Basalt fibers epoxy resin composites possess very high mechanical properties, higher than glass fibers composites and similar to carbon fibers composites. The tensile modulus for $\pm 45^\circ$ and $0^\circ/90^\circ$ laminates is equal to 11.3 GPa and 23.9

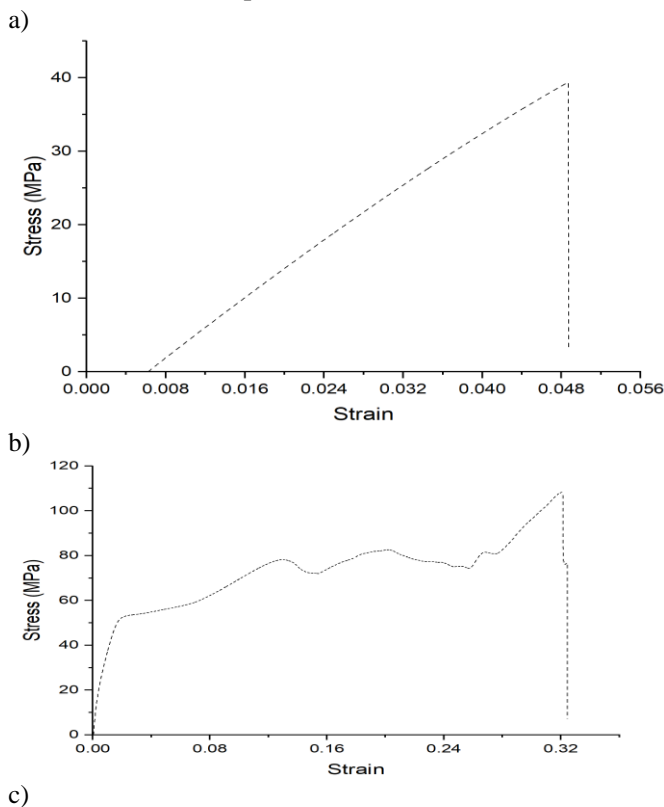
GPa respectively. The UTS value shows the huge increasing thanks to contribution of the fibers in the epoxy resin matrix.

Reference

[1] ASTM D3039 / D3039M-17, Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials, ASTM International, West Conshohocken, PA, 2017, www.astm.org

[2] ASTM D3518 / D3518M-18, Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a $\pm 45^\circ$ Laminate, ASTM International, West Conshohocken, PA, 2018, www.astm.org

[3] Dorigato A, Pegoretti A. Fatigue resistance of basalt fibers-reinforced laminates. *Journal of Composite Materials*. 2012;46(15):1773-1785. doi:10.1177/0021998311425620



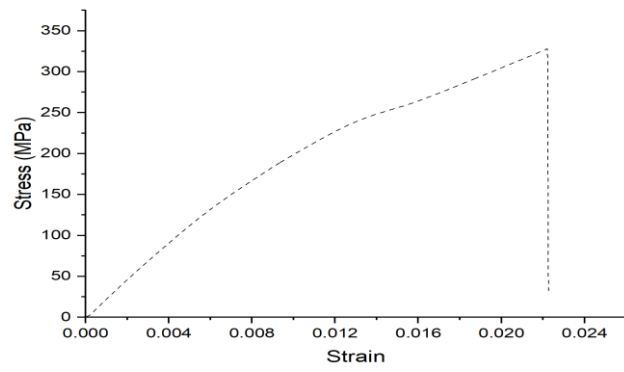


Fig. 1 – Stress and strain for a) epoxy resin, b) $\pm 45^\circ$ laminates, c) $0^\circ/90^\circ$ laminates.

