Mechanical properties of basalt fiber/ epoxy resin composites

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Abstract: *This paper presents a study regarding the obtaining, characterizing and mechanically testing a new laminar composite material, consisting of epoxy resin reinforced with basalt plain fabric.*

The composites were obtained by manual lay-up, cross-link stage being developed by pressure molding in a hydraulic press. Rectangular plates were obtained and cut into samples with specific shapes, for the mechanical tests (tensile, three-point bending and compression). After testing, the fracture zone was analyzed using optical microscopy to observe the behavior of the composite following the mechanical stresses applied (fracture mechanism, voids presence and fiber delamination identification).

Due to the low costs and non-hazardous nature, basalt fibers can be a serious competitor in the production of laminar composites that could successfully replace ordinary glass fiber composites. The mechanical properties in tensile, three-point bending, and compression of epoxy-resin-impregnated basalt fiber composites are comparable and even exceed those of widely used epoxy-resin-impregnated fiberglass composites.

Key Words: *3D printing, tensile testing, optical investigation, SEM analysis*

1. INTRODUCTION

The new trend for obtaining composite materials is to use ecological and circular components, with low environmental impact. Natural fibers, including mineral and vegetable fibers, are increasingly used for polymer composite materials due to their versatility and easily processable [1]. Due to the low costs of the production processes, the use of natural/ ecological fibers as reinforcement for the manufacture of lightweight polymer composites, with reduced costs, exhibits a global tendency of increase [2].

One of the most well-known thermosetting polymers, epoxy resin, is the preferred polymer matrix in applications aimed at electrical insulation, coatings, laminates and composites due to its outstanding advantages: high thermal conductivity, low shrinkage, mechanical stability, easy handling, excellent chemical and physical resistance [3, 4].

In an environment where climate change has accelerated the search for sustainable, eco-friendly polymer composites, by balancing the requirements of performance, durability and safety, the use of materials with recirculation (recyclability) takes precedence [5].

The most common fibers used as reinforcement in composite materials are glass fibers (GF) and respectively carbon fibers (CF). Efforts to control environmental problems and reduce processing costs have led to the study of alternatives to replace these fibers, to the creation of durable composites with natural mineral fibers. Basalt fiber is one of the important mineral fibers produced from volcanic rock. The resulting compounds considerably reduce the weight and final cost of laminated composite materials and their properties are comparable to their replaced predecessors [6, 7]. Basalt fibers are cost effective and offer exceptional properties (Table 1) [1] compared to glass fibers. Both mechanical (high Young's modulus and strength, hardness, durability) and thermal (high temperature applications) properties make basalt fiber composites (BFRP) to be studied with increased interest recently. Unlike fiberglass plastic, the high crystallizing ability of basalt fibers greatly simplifies the recycling of composite materials [8]. Basalt fibers have equivalent tensile strength, higher modulus and better alkaline resistance than glass fibers, and compared to carbon fibers, they have better fireproofing and thermal insulation properties [4, 9].

Raw basalt fibers are emerging green mineral fibers based on natural environmentally friendly ore (igneous rocks) with superior thermo-mechanical properties to commonly used glass fibers [10]. Their production process requires no new components addition and involves no hazardous substances emissions (such as boron or alkali metal oxides, as the case with glass fibers), resulting in their composition being the same as the basalt ore, therefore further degradation after its end of use generates no environmental pollution as they can safely be returned to the soil as a sustainable material [11]. Beyond this, due to its oxidation resistance, it can act as efficient catalyst carrier in wastewater treatment and gas purification, without secondary pollution [12].

Given their sustainability on all levels (composition, production, degradation), they represent a compatible candidate to be replace glass fibers in composites, as when used with recyclable matrix, they can results in alternatives that can be easily managed into new production/usability cycles and contribute to the circular economy flows.

Table 1. Comparison of mechanical properties between basalt and glass fibers [1, 13]

Properties **Fibre Type**	**Basalt**	**E-glass**	**S-glass**
Basal diameter (μm)	6-21	6-21	6-21
Density (g*cm^{-3})	2.65-3.00	2.55-2.62	2.46-2.49
Tensile Strength (MPa)	3000-4840	3100-3800	4590-4832
Modulus of Elasticity (GPa)	79.3-93.1	72.5-75.5	88.-91
Elongation at Break (%)	3.1	4.7	5.6
Price (USD*Kg^{-1})	2.5-3.5	0.75-1.2	5-7

Basic mechanical and thermal properties of basalt fibers compared with other commercial fibers presented in Table 1 show their excellent thermal properties and tensile strength and good modulus. Adding the significant advantage of being eco-friendly, nontoxic, lightweight and easily affordable, they are ideal candidates for fabricating composites for several applications [2, 14]. Several studies attest the tailoring of mechanical and impact properties of basalt fibers composites having as matrix both thermosets (vinylester, epoxy, phenolic) and thermoplastics (polypropylene, polyethylene, polylactic acid) [2, 10, 14, 15, 16, 17]. Some of them used surface treatment and/or coupling agents attached to the fiber surface to improve the chemical/physical interaction with the polymer, some combined basalt with glass, carbon or aramid fibers [2].

Basalt fibers are successfully used in civil infrastructure and construction applications, infrastructural industry, corrosion resistance in chemical industry, wear, friction and high temperature-insulation in automotive, anti-low velocity impact target area, fire protection and resistance applications [2].

In general, the mechanical properties of laminar composite materials depend on the mechanical properties of the constituent materials, the adhesion strength between the fiber and the matrix, the nature of the interfacial bonds and the load transfer mechanisms in an interface [8]. The reinforcing elements as well as the polymer matrix that constitutes the composite material influence the mechanical properties of the composite materials. Studies have shown that the manufacturing technology of these materials is an important factor that can ultimately vary/influence the numerical values of the mechanical properties [18].

In order to improve these mechanical properties, studies in this regard were carried out by modifying the polymer matrix (epoxy resin) by adding nano or micro-metric particles (graphene nanoplates, nanoclay particles, graphene nanopellets, multi-walled carbon nanotubes, etc) [19, 20, 21, 22, 23, 24], but also by treating the surface of the basalt fiber fabric with silanes [5, 25].

Depending on the orientation of the two-dimensional fibres and the placement of the layers in the structure of the layered composite material, the mechanical behavior and the breaking/yielding morphology after mechanical testing indicate different properties and behaviours [26, 27, 28].

Compared to traditional composites, reinforced with glass or carbon fibres, high-performance composites based on basalt fibres are becoming more and more available, although their mechanical properties are currently still to be exploited and discovered [29].

Because of its higher heat resistance, superior ductility, and lower cost, they prompted Guangyong Sun et al. to study, experimentally, analytically and numerically, the mechanical behavior of hybrid composites formed by combining carbon and basalt fibres. The obtained results indicated high, improved values, and the analytical model as well as the numerical results are validated against the experimental results [30].

Fiberglass/basalt hybrid composites in various configurations exhibit additional impact advantages [31].

And to improve the rigidity and thermal resistance of basalt fiber reinforced laminar composites, the addition of basalt powder makes a positive contribution, the numerical values of these properties increase [32].

In the current study, a laminated composite material was obtained by manually impregnation of 12 layers with L20 epoxy resin, layer by layer, with curing under pressure of $5kg/cm^2$, in a hydraulic press (manual lay-up compression molding). Specimens with specific geometric shapes were cut from the composite plate and mechanically tested in tension, 3-point bending and compression. The mechanical properties were analysed, as well as the mode of yielding by fractography, in order to observe the behavior of the 12Bas/Epoxy composite, following mechanical stresses.

2. EXPERIMENTAL

2.1 Materials

The resin-hardener system used as a matrix was based on Epoxy Resin L20 and EPH 161 hardener, purchased from R&G Faserverbundwerkstoffe GmbH, in 100:25 by weight mixing ratio, according to the manufacturer recommendations.

The reinforcement material used consisted of plain type Basalt fabric, purchased from Basaltex-Flocart NV, Wevelgem, Belgium. The main properties of these components are presented in Table 2.

Table 2. Materials main properties

Materials	Properties/ Specifications	
Epoxy resin L20/Hardener EPH 161	Reaction product	Bisfenol-A-(epichlorhydrin)
	Density	1.15 g/cm^3
	Viscosity	900 mPa*s (25°C)
	Flash point	>120 °C
Basalt fiber	Weave type	plain
	Specific surface weight	220 g/m^2
	Melting point	1350 °C
	Sizing type	silane

2.2 Processing and testing methods

The technology used for the development of layered composites was manual lay-up, consisting of manual impregnation layer by layer and curing (cross-linking) of the resin under pressure. 12 layers of basalt fabric were manually impregnated with epoxy resin/ hardener mixture and it was subjected to curing under pressure in a hydraulic press, using a pressing force of 5kg/cm^2, the curing reaction taking place under pressure over 24 hours, as the manufacturer indicated.

The composite plate obtained was cut into specific shapes for mechanical tensile, three-point bending and compression tests, according to international standards (fig. 1).

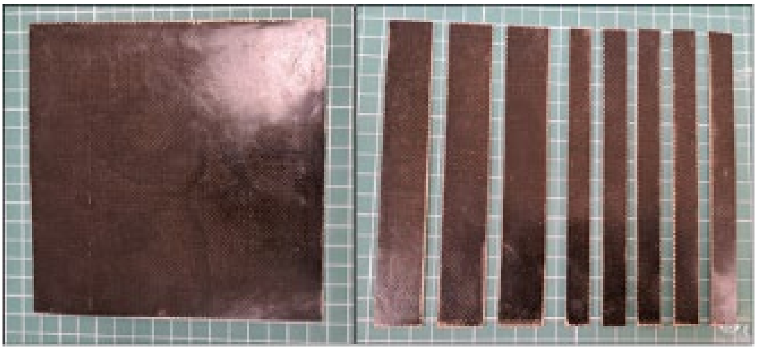

Fig. 1 Laminar composite Basalt Fiber/Epoxy L20 (12Bas/Epoxy) dimensioned and cut to the specific shapes of mechanical tests

3. RESULTS AND DISCUSSIONS

3.1 Mechanical tensile testing

To evaluate the mechanical properties, the 12Bas/Epoxy laminated composite was subjected to mechanical testing using the INSRON 5982 machine equipped with a 100kN force cell and an optical extensometer with a 200 mm field of view. All mechanical investigations were carried out in an ambient environment, with constant temperature and humidity.

A number of 3 specimens were tested for each individual test and the average values and standard deviation of the mechanical properties were listed in Table 3.

3.1.1 Tensile testing

The tensile testing was carried out according to the international standard ISO 527-4 Type 3. The testing speed used was 5 mm/min with a preload of 10N.

According to the ISO 527-4 standard, laminar composite samples must have tabs added to the upper ends of the samples, to avoid the appearance of imperfection areas during clamping between the grips of the testing equipment. In fig. 2 you can see the tabs caught over the samples, caught in the grips of the equipment and failure zone for samples from composite material 12Bas/Epoxy.

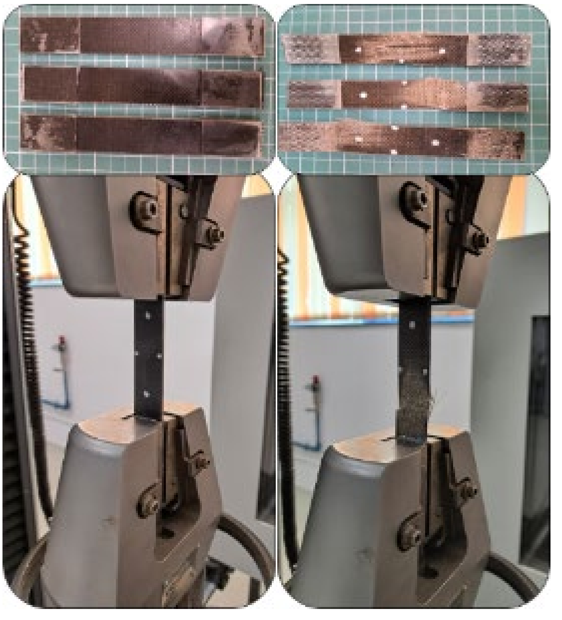

Fig. 2 The samples for tensile testing and placing between the grips of the equipment

During the mechanical tensile testing, the materials exhibited a standard brittle material like behavior, the breaking area is between the visualization points of the optical extensometer, failure mode was according to the international standard ISO 527-4. The breaking mode will be analyzed in detail with optical microscopy to observe the behavior of the composite.

Fig. 3 shows the graph stress-strain curve of 12Bas/Epoxy composite materials after tensile tests.

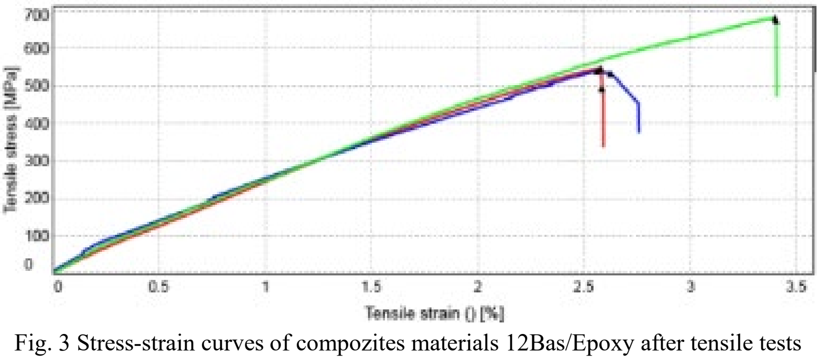

Fig. 3 Stress-strain curves of compozites materials 12Bas/Epoxy after tensile tests

For the mechanical tensile testing, the behavior trend is similar for all tested samples, the values of the averaged mechanical properties are illustrated in Table 3.

3.1.2 Three point pending tests

The 3 point pending tests was carried out according to the international standard ISO 14125 3-point bending class IV. The testing speed used was 2 mm/min with a preload of 0.1N.

The geometric shape of the samples for the 3-point bending test is rectangular, and the fixed support span on which the samples are placed were positioned with a distance of 80 mm between them (fig. 4).

Fig. 4 The samples for 3 point bending tests and placing between the grips of the equipment

Also in this case, during the bending testing, the materials exhibited a standard brittle material like behavior. the breaking zone will be analyzed in detail with optical microscopy to observe the behavior of the composite.

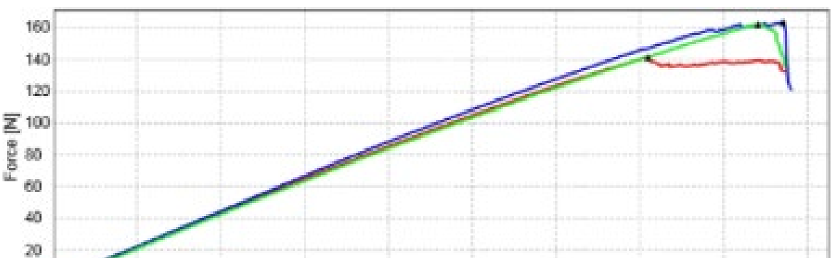

Fig. 5 Force-strain curves of compozites materials 12Bas/Epoxy after 3 point bending tests

For the mechanical 3 point bending tests, the behavior trend is similar for all tested samples, the values of the averaged mechanical properties were illustrated in Table 3.

3.1.3 Compression tests

The compression tests was carried out according to the international standard ASTM D 6641M. The testing speed used was 1.3 mm/min with a preload of 1N.

The geometric shape of the samples for the compression test is rectangular and the clamp is made between 4 metal blocks, shown in the fig. 6, which keep it fixed during the entire period of the test.

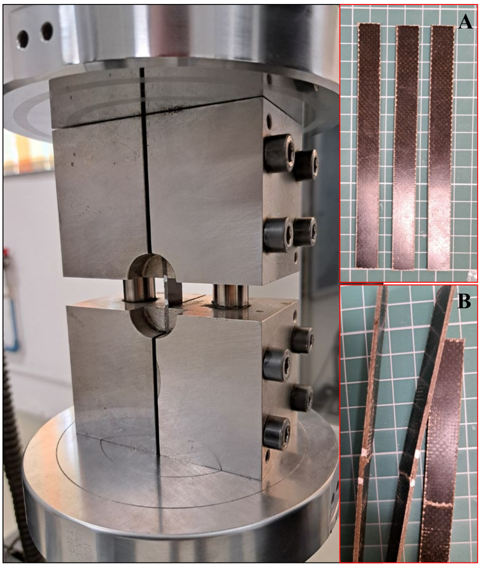

Fig. 6 The samples compression tests and placing between the grips of the equipment

The materials exhibited a standard behavior during compression testing, the breaking area is between the visualization points of the optical extensometer, the failure mode according to the standard. The failure mode will be analyzed in detail with optical microscopy to observe the behavior of the composite.

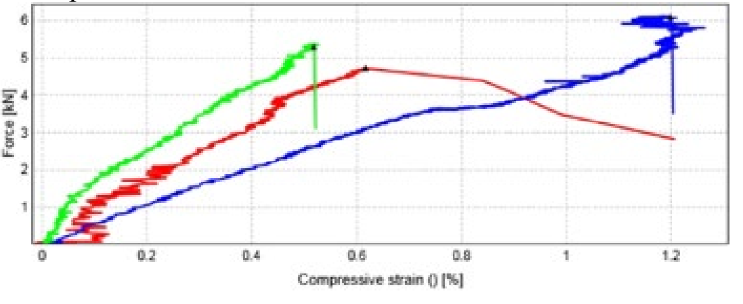

Fig. 7 Force-strain curves of compozites materials 12Bas/Epoxy after compression tests

For the mechanical compression tests, the behavior trend is similar for all tested samples, the values of the averaged mechanical properties were illustrated in Table 3.

Table 3 presents the averaged numerical values of the investigated mechanical properties for the studied laminar composite.

Table 3. Mechanical properties of basalt laminated composites (12Bas/Epoxy samples)

Mechanical testing	**Force [kN]**	**Modulus [GPa]**	**Strength [MPa]**	**Elongation [%]**
Tensile	22.5 ± 3	28.11 ± 3.6	589.54 ± 78.9	2.84 ± 0.09
3-point bending	0.16 ± 0.01	29.82 ± 1.7	412.36 ± 42.3	1.62 ± 0.17
Compression	5.4 ± 0.69	41.84 ± 17	255.76 ± 26.8	0.81 ± 0.4

It can be observed that the numerical values obtained for all the specimen batches exhibit low standard deviation values in general, indicating the compact structure of the layered composites suggesting uniform resin distribution and fiber placement was achieved in bulk and on the entire surface of the composites. Compared to similar epoxy composites reinforced by glass fiber fabric manufactured using the same method (manual lay-up and pressing), the basalt reinforced composites developed exhibit superior mechanical characteristics in terms of tensile, 3-point bending and compression testing [33, 34].

3.2 Fractographic analysis

The fracture mode following different mechanical tests of the of samples basalt-based laminar composite material was analyzed by observing the fracture area of the samples through optical microscopy. The MEIJI 8520 microscope equipped with a video camera was used to capture optical microscopy images for the samples, using a 40x magnification level.

The images were captured in the failure area of the sample after the mechanical tests, and highlighted the mode of failure for each mechanical test separately (Fig. 8- Fig. 10). Fig. 8 illustrates the fracture area of two of the specimens tested in tensile loadings. The first specimen fractured due to fiber strain, the fracture area showing a transverse direction crack along the specimen width. The optical micrographs in Fig. 8 (a) and (a’) are in accordance with the visualized failure mode, the middle area of the layered structure maintained its integrity, while lateral layers debonded, as shown by the cracks that propagated along the length of the specimen over 1-3 mm. The specimen 2 failure mode shows a fracture causing more visible and dramatic damage to the layered structure. Besides the fracture along the width of the specimen, the fibers completely delaminated in the fracture area as shown by Fig. 8(b), with crack propagating on extended lengths along the specimen as shown by Fig. 8(b’).

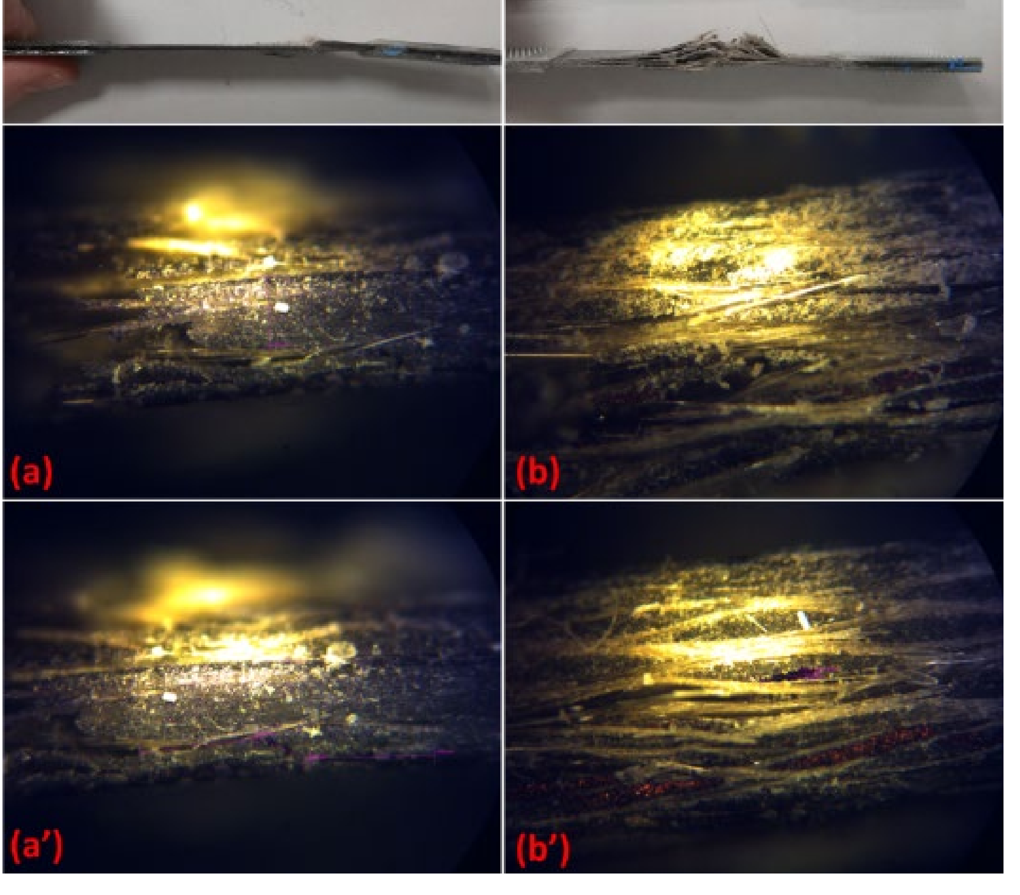

Fig. 8 Optical micrographs of the fracture area in two specimens tested in tensile loadings (a) and (a’) Specimen 1, (b) and (b’) Specimen 2

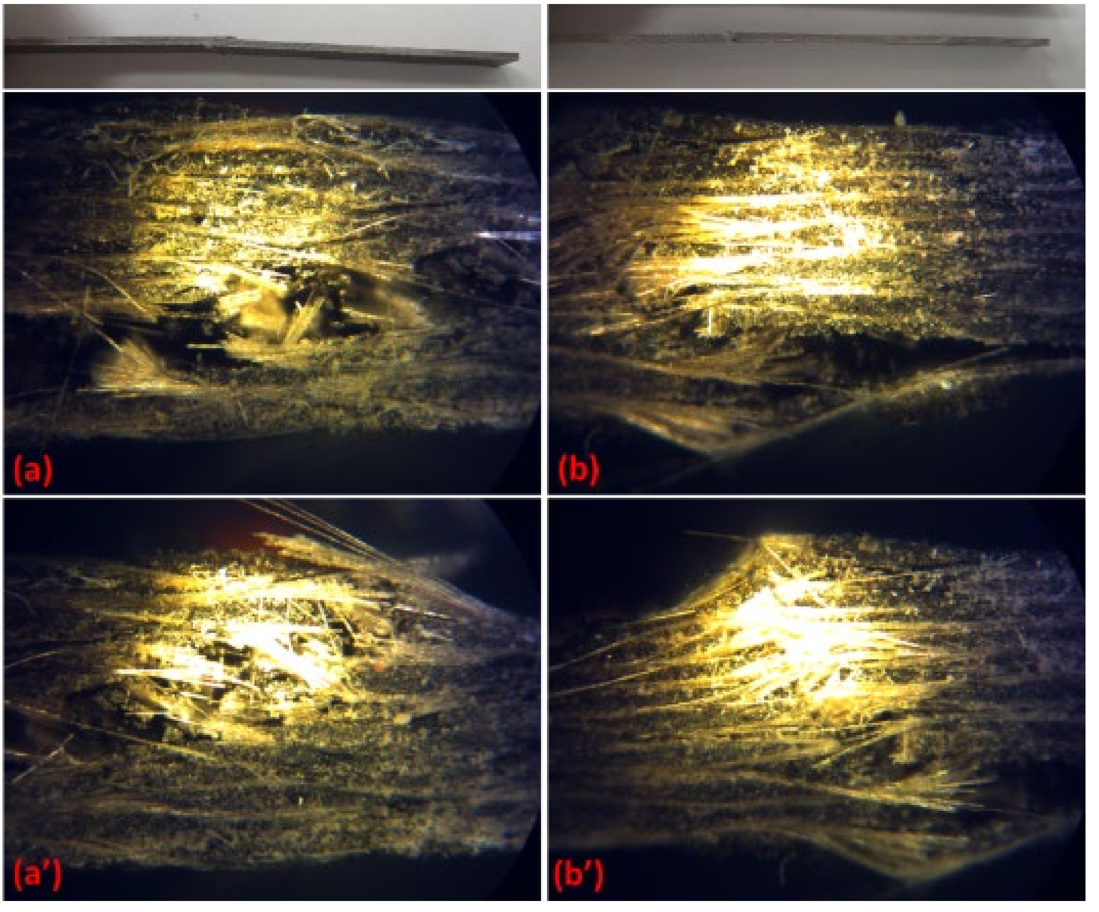

Fig. 9 Optical micrographs of the fracture area in two specimens tested in compression loadings (a) and (a') Specimen 1, (b) and (b') Specimen 2

Fig. 9 illustrates the optical micrographs of the fracture area visualized in two of the specimens tested in compression.

In both specimens, it can be observed that the materials failure occurred sidelong according to the standard failure modes in compression. The fiber and matrix breaking area is limited to the maximum loading application region, crack propagating phenomenon occurs only in adjacent areas.

Fig. 10 illustrates the optical micrographs of the fracture area visualized in two of the specimens tested in 3-point bending loadings.

On a macroscopic level, the specimen seem to have suffered the fracture of one external layer only, however, the microscopic images show that in both specimens, the bending stresses generated cracks in the layered structure of the composite that propagate between 0.3 and 1 mm length, as shown in Fig. 10 (a), (b), (b').

However these cracks presence is only visualized in a limited area, around the load application vector, and they do not propagate in further away areas.

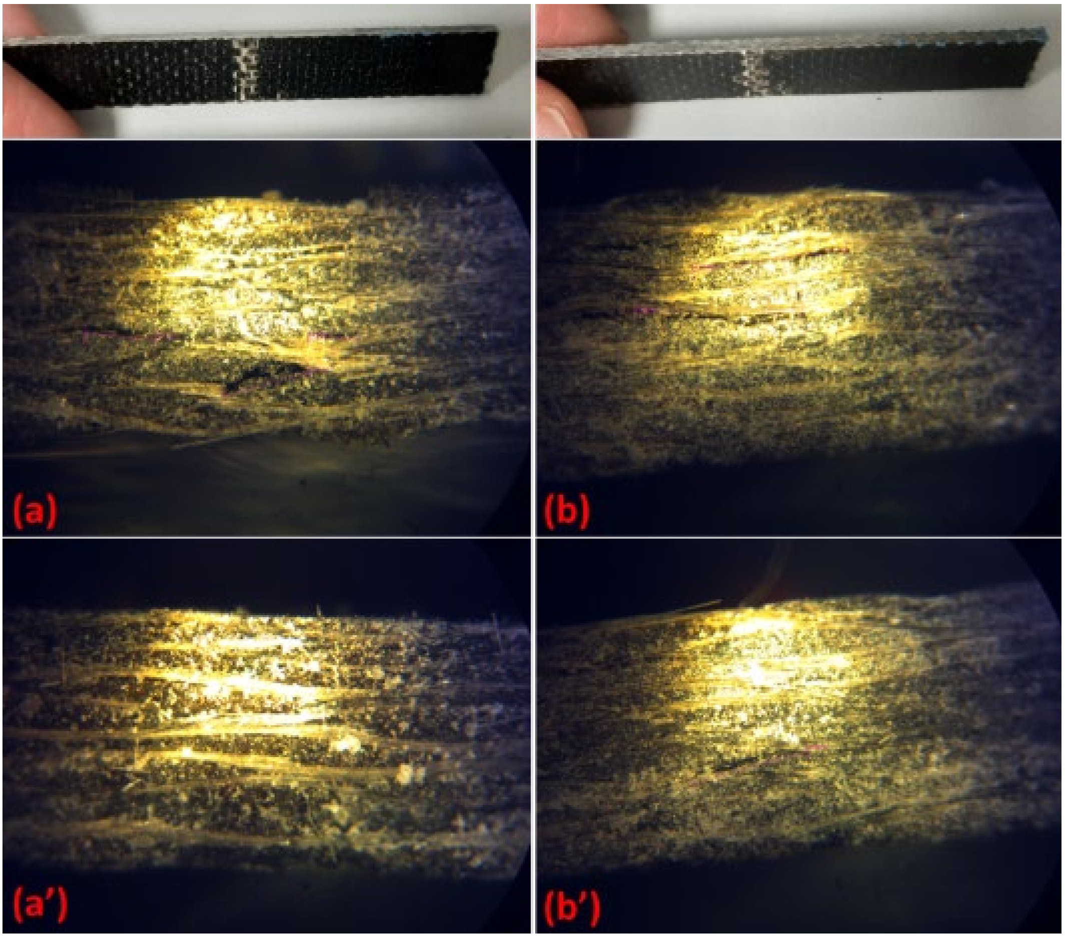

Fig. 10 Optical micrographs of the fracture area in two specimens tested in 3-point bending loadings (a) and (a') Specimen 1, (b) and (b') Specimen 2

Optical micrographs did not show the presence of voids or other structure defects in any of the visualized specimens, indicating that the manufacturing methods and parameters led to materials with optimum microstructure from mechanical point of view.

The optical microscopy images of the fracture area, confirms that the layered composites withstood successfully the applied forces in the different loading types tested, supporting the observation that the basalt fibers/epoxy composites can act as high performance composites, exhibiting failure modes according to the accepted standards.

4. CONCLUSIONS

The study presents the development and mechanical testing of basalt fiber fabrics reinforced epoxy composites, highlighting the behavior of these materials when subjected to tensile, compression and 3-point bending stress.

Mechanical test results indicate that these materials can act as a potential candidate to replace the standard glass fiber reinforced epoxy composites, in the path to use more non-hazardous, environmental friendly and circular materials in fiber reinforced composites sectors. Their mechanical performance is similar to that of glass fiber reinforced epoxy, while using natural source, non-hazardous reinforcement agents.

The optical microscopy analysis illustrated the fracture mode, after the mechanical tests, of the 12Bas/Epoxy composite laminate samples, and the behavior following the fracture was observed. The optical microscopy images of the fracture area, confirmed that the layered composites withstood successfully the applied forces in the different loading types tested, supporting the observation that the basalt fibers/epoxy composites can act as high performance composites, exhibiting failure modes according to the accepted standards. Although these materials are highly anisotropic by nature, as all composite materials, the specimens exhibited similar for each individual mechanical stress performed, confirmed both by the similar values of strength, stiffens and strain as well as by the similar fracture modes and uniform microstructure visualized in all specimens.

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