ADHESION TEST OF RESIN-INFUSED BASALT FIBERS FOR WIND ENERGY APPLICATIONS

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Abstract

Rotor wind blades are principally made of E-glass fibers reinforcement into an epoxy resin. This fibers have already reached the limit of their mechanical properties, therefore new materials are needed. On one hand, carbon fibers offer a higher level in both stiffness and strength, but come at a significantly higher price. Basalt fibers, on the other hand, represent a viable alternative to E-glass, with better mechanical properties at a comparable price.

The strength of basalt fibers can be fruitfully used in wind energy applications if the strength of the fibers is transmitted to the matrix. Therefore, the fiber-matrix-adhesion is a crucial parameter that characterize the composite's quality. The aim of this paper is to present the possibility for different tests methods concerning fiber-matrix-adhesion.

1. Introduction

1.1. Basalt fibers

Basalts are rocks formed from the rapid cooling of magma from volcanic eruptions, therefore it'san inorganic material, a silicate composed by a mix of different oxides (SiO2 45 % - 52 %, Al2O3 12 % - 16 %, FexOy 6 % - 18 %, alkaline earth oxides 10 % - 20 % and alkaline oxides 2 % - 8 %).
In the last decades different types of basalt have been successfully fiberized. These fibers are nowadays manufactured by a melting process.
The chemical composition of basalt fiber strongly differs depending on the geographical location of the raw materials. Due to the composition variability of natural rocks, basalt fiber production is still an issue. The fiberization process is related to the viscosity and the temperature. Basalt has a short temperature range fitting the viscosity requirements.

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Basalt fibers are stable under thermal treatment and alkaline leaching when compared to the benchmark of the E-Glass fiber. They have high strength (up to 4,84 GPa) and stiffness (up to 110 GPa).
Due to their chemical, thermal and mechanical properties basalt fibers are applicable in different textile products. The main applications are as reinforcement of concrete and of plastics.
The basalt fiber production reaches 30.000 tons and it represents a niche product in comparison to glass fibers (more than 4 million tons in 2011 [1]).
There are only a few basalt fiber producers around the world: Kamenny Vek (Russia), DBF – Deutsche Basalt Faser GmbH (Germany), Technobasalt (Ukraine), GBF Basalt Fiber Co. and Hebei Tong Hui Science Technology Co. (China), Isomatex (Belgium) and Mafic (Ireland). [2]

1.2. The need for a standard method to determine the influence of sizing on basalt-fiber-matrix-adhesion

The strength of basalt fiber reinforced plastics is manly influenced by three factors: the mechanical properties of the fibers, the mechanical properties of the polymeric matrix and the properties of the interface between the basalt fiber and the polymeric matrix. The first two elements can be easily proved and adapted separately, while the properties of the boundary layer between the fibrous reinforcement and the matrix are related to the interaction between this two materials.
The most relevant property of this interlayer between matrix and fiber in composite materials is the fiber-matrix adhesion. This property is quantified by the measurement of the interlaminar shear strength,that represents the maximum force which can be transferred from the fiber to the matrix. The interlaminar shear strength is a shearing stress and is measured in N/mm^2.
Low interlaminar shear strength indicates low adhesion between fiber and matrix, therefore the fibrous material will unbound from the matrix when the fiber reinforced plastic is under stress and consequently, the fibers won't be able to transmit the stress to the matrix and to the composite.
The improvement of the fiber-matrix-adhesion is crucial, however the value of the interlaminar shear strength is important to have a benchmark parameter for evaluating improvements in adhesion properties.
In order to improve the adhesion the basalt fibers are subjected to a surface treatment during their production. After the fibers formation, a sizing is applied to the surface of the filaments to. prevent from damages, in particular when the fibers are manufactured into textile structures [3]. For composites applications, the sizing also increases the adhesion properties.
The surface of basalt fibers is chemically nearly inert and the fiber-matrix-adhesion is poor. Therefore, different coupling agents (e.g., silanes) have been developed and integrated into the sizing.
Detailed analysis of the boundary layer between basalt fiber and matrix are as necessary as the mechanical properties of each material (basalt fiber and matrix).

2. Interlaminar shear strength: Testing methods

Numerous analysis methods have been developed to measure the interlaminar shear strength between fiber and matrix in composites. Theinterlaminar shear stress is always combined with other forms of stresses, such as tensile stress, pressure and torsion. Even if the quality of the composite depends significantly on the interlaminar shear strength, its determination independently from other stresses is a great issue.
In general, testing methods for the determination of the Interlaminar shear strength are divided into two clusters: direct and indirect testing methods (see Figure 1).

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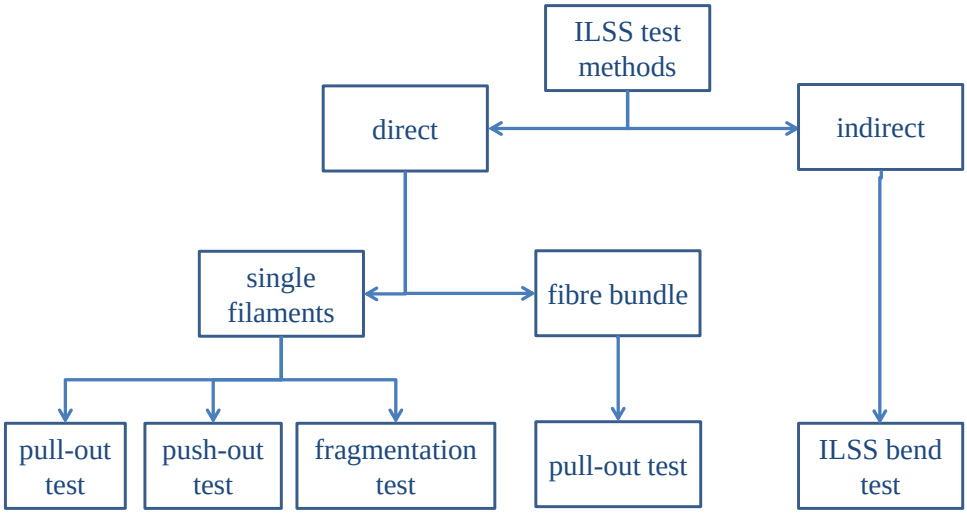

Figure 1: Overview about different test methods for the interlaminar shear strength (ILSS)

By indirect testing, the final result is not measured but calculated. By direct testings, the test samples are stressed in a way that the unidirectional stress allows a direct calculation of the interlaminar shear strength. However, local deviances from this unidirectional stress might occur within the direct testing too. Between the direct methods, fiber bundle pull-out-tests present several issues related to the accurate determination of the surface subjected to the unidirectional stress and the homogeneity of the applied forces to each filament. The direct testing on single filaments (pull-out-test, the push-out-test and the fragmentation test) are the most valuable tests. Those last methods are based on the Eq. 1, in which the interlaminar shear strength is measured directly by determining the tensile force applied to pull or push out the fiber from the matrix, knowing the cylindric surface between fiber and matrix:

$$\tau = F / A = F / (\pi \cdot d \cdot l) \quad (1)$$

In Eq. 1, τ represents the interlaminar shear strength, F is the applied force and A is the boundary surface between fiber and matrix. This area can also be expressed by the diameter d and length l of the fiber. In Figure 2 is represented the pull-out principle of a single filament that is embedded into a thin matrix block. During the measurement, the fiber is pulled out of the matrix by applying a force F until the fiber separates from the matrix. The interlaminar shear stress is determined by the extreme force which is measured before unbounding [4]. By determining the interlaminar shear strength by pull out test, will be supposed that only the shear stress in the direction of the fiber occurs. Actually, at the interlayer between fiber and matrix also a normal stress can occur. As an alternative to the pull out test, there is the push out test. This test method can be calculated in the same way as the single filament pull-out-test (Eq. 1).

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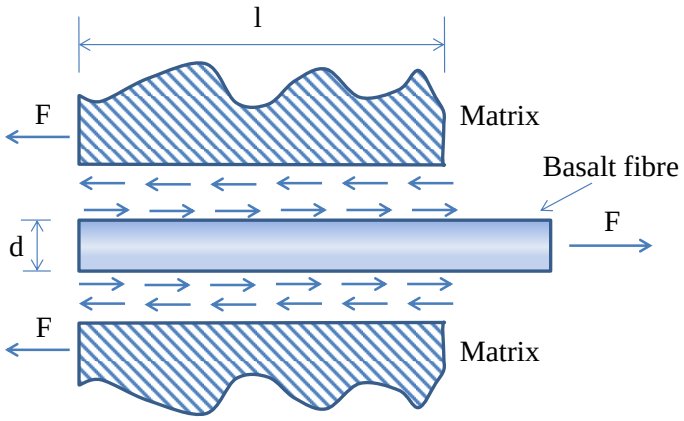

Figure 2: Interlaminar shear stress (ILSS) between fiber and matrix. [5]

The last suitable method in Figure 1 for estimating fiber-matrix-adhesion is the single filament fragmentation test, in which a single filament is completely embedded into a matrix sample. This sample, well polished, will be put under a microscope and stretched. During the elongation of the sample, the incorporated filament will be also elongated. The shear stress in the boundary layer between matrix and fiber will be considered constant. The sample will be stretched with constant elongation's rate. Because of the increasing stretching, the tension in the filament will be also increasing until it comes to the filament break (see Figure 3).

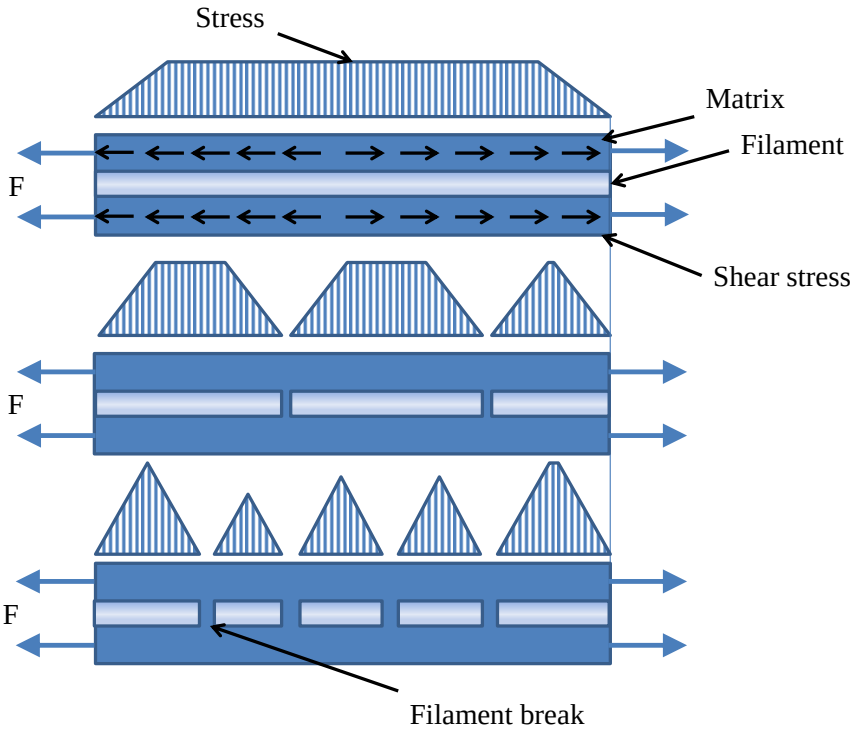

Figure 3: Tension distribution during the single filament fragmentation test [5]

The basalt fiber has an elasic and brittle behavior. It, as well as glass fibers, breaks in presence of fiber defects, whichwill be statistically distributed on the fiber length. As shown in Figure 3, the tension decreases to zero along the filament at each fragmentation's ends.

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By increasing the elongation and the stretching, the number of fragmentations in the fiber increases until a saturation point. This testing system is based on the principle that the shear stresses between fiber and matrix transfers tensile stresses in the basalt fiber itself. If the tensile stress exceeds the strength of the basalt fiber, the fiber breaks into two parts. Once the fiber parts are too short, the shear stresses cannot induce enough tensile stress into the fiber so that it will not break again. The interlaminar shear strength τ can then be calculated using the following equation 2:

$$\tau = \frac{\sigma_z \cdot \mathrm{d}}{2 \cdot \mathrm{l}_c} \tag{2}$$

σ_z is the strength of the basalt fiber filament. σ_z was determined by measuring the strength of 50 single filaments without matrix, e. g. with a Favimat testing unit.
d is the diameter of the fiber and l_c is the critical length of the broken fiber pieces. L can be derived from the mean length of the fiber pieces by multiplying with 4/3. This formula is derived in [6]

A crucial aspect of this procedure is to reach fragment saturation. Another method of ensuring that the saturation level is reached is to pre-strain the fiber byattaching weights at the ends of the fibers before embedding them in the epoxy resins. Pre-straining also accounts for the compressive thermal residual stresses induced in the fiber due to resin curing. The formulas for calculating the thermal residual stresses and thus for the necessary pre-strain are also derived by Feih et. al. [6]

Procedure and Apparatus
The conducted research involved the following steps: silicone mold preparation, basalt fiber sample preparation, microscope measurements and final image analysis.

Commercial basalt fibers were provided for the analysis by Kammeney Veg. This product uses sizing for better performance with epoxy resin. The diameter of the fibers and their mechanical properties were previously determined.
Due to the higher strain-to-failure values of 3.2% of the basalt fibers, compared to 2% for carbon fibers, two methods for preparing the fragmentation samples were used. The first method involved no pre-straining of the fibers. For the second method, the fibers were pre-strained.
Firstly, the samples were polished using a polishing machine. However, this led to a non-uniform thinning of the samples, thus compromising the measurements. Following, the samples were polished by hand using 800, 1200, 2500 and 4000 grit polishing paper.

The polished samples were fixed in the tensile tester and set on an XY-Table under an optical microscope with a 25x magnification. The samples were strained by turning the micrometer-screw of the tester (Figure 4) for 90^0 until the first break and then for 180^0 until fragment saturation or sample break occurred. For each strain-level, two sets of pictures were taken using regular light and polarized light to observe the stress field around the fiber breaks.

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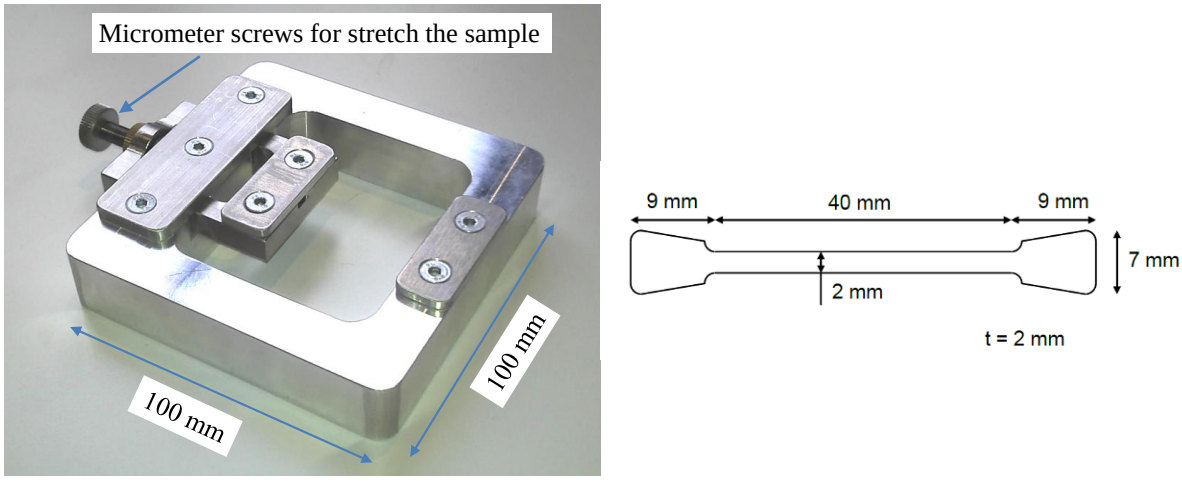

Figure 4: Tenter (left) and sample geometry (right)

The pictures were then stitched together using a CorelDRAW macro previously written. The analysis of the total elongation of each fiber and the number of fiber breaks was performed by using an ImageJ macro.

4.1 Quantitative Discussion

The Basalt fibers failed to reach a saturation point before occurred a necking in the sample. Thus saturation happened outside of the linear-elastic region of the epoxy matrix and in some cases it was not reached even at the sample break. Because of this, the Interlaminar shear stress values could not be fully determined. Graph of the number of breaks until necking vs. strain are showed for the pre-strained basalt fibers in Figure 5.

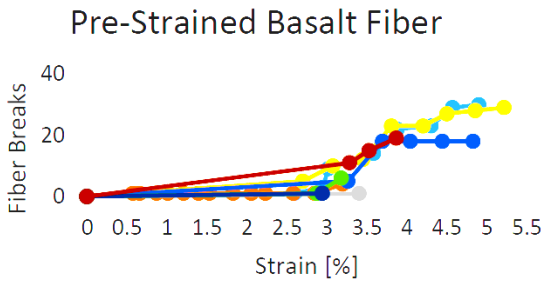

Figure 5: Fiber breakes vs. strain in basalt fiber

4. Conclusions

In this paper, the need for a quantitatively investigation of the quality of fiber-matrix-adhesion was illustrated and different methods to determine the interlaminar shear strength, ILSS, were presented. The single filament fragmentation test was considered to be the most suitable method in order to investigate the processes at the fiber-matrix-interface so the reasons for the breakdown could be understood by applying this test. By the first reasonable results, the single filament fragmentation test can be considered a suitable method also for basalt fibers.
Even if the single filament fragmentation test showed a high potential, the method still requires modifications in order to be adapted to basalt fibers. In order to determine the adhesion between fiber and matrix, it's necessary to reach the saturation of fibers breaks. The most feasible adaption would be

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to apply a higher pre-straining so the strain in the composite will be reduced from 3.2 % to 2 % by the saturation point, preserving the sample to failure before the saturation of breaks.

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