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Mechanical properties of basalt fibers and their adhesion to polypropylene matrices

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ABSTRACT

This work is aimed to study the mechanical properties of basalt fibers, and their adhesion to polypropylene (PP) matrices. Single filament tensile tests were used to calculate the strength of different types of fibers, characterized by different providers and surface treatment. Single fiber fragmentation tests (SFFT) were used to calculate the critical length of the fibers, in a homopolymer PP matrix and in a maleic anhydride modified PP matrix. It was shown that the tensile strength of the fibers is not significantly influenced by the origin or the surface treatment. Only fibers without any sizing show very reduced mechanical properties. On the other hand, the tensile strength was shown to be severely dependent on the filament length. Weibull theory was used in order to calculate the fitting parameters σ_0 and β, which were necessary in order to extrapolate the tensile strength to the critical length determined by SFFT. This allowed calculating the adhesion properties of the basalt fibers. It was shown that fiber–matrix adhesion is dependent on both the presence of sizing on the fiber surface, as well as on the modification of the matrix.

1. Introduction

In the last years, the increasing interest in environmental issues has promoted the use of natural fibers in polymer based composites [1,2]. Many types of natural fibers have been studied, including vegetal fibers, which are, however, very sensitive to thermal and hygroscopic load and show limited mechanical properties. A possible solution which overcomes the disadvantages of vegetal fibers, though taking into account the environmental issues, is represented by the use of natural mineral fibers, like basalt.

In fact, basalt fibers are spun at high temperatures from selected melted basalt rocks, and their production does not require the addition of secondary raw components, as is the case of glass fibers [3]. Therefore, in the last years continuous or short basalt fibers have been studied in view of their potential applications in polymer matrix composites [4–8]. Based on the specific nature of the rock, different categories of basalt fibers, with different chemical compositions and mechanical properties, can be obtained. The last generation of basalt fibers is characterized by excellent sound insulation properties, heat resistance (higher than glass), and good resistance to many chemicals [6]. Compared to vegetal fibers, basalt fibers are characterized by excellent mechanical performances, similar, or in some cases even higher, to those of glass fibers [6]. On the other hand, the cost of basalt fibers, though higher than that of E-glass, is lower than the cost of S-glass, making this material suitable to replacement of glass fibers in various industrial fields. Besides the applications of basalt fiber in polymer matrix composites, there are other new application fields as, for example, bionic honeycomb panels and the wood–plastic composites [9–12].

With particular attention paid to thermoplastic matrix composites, different studies showed the potentiality of such fibers as substitutes of glass fibers [5,7,8,3]. Nevertheless, it was shown that in order to attain an efficient reinforcement, the adhesion properties between matrix and basalt fibers should be improved. This can be done either by surface modification of the fibers, which can be accomplished by thermal or chemical treatment [13,14], or by bulk modification of the polymer matrix, which involves addition of different types of compatibilizers, among which maleic anhydride grafted polypropylene (PPgMA) has been the most widespread used [5,8]. In the case of basalt fibers, thermal treatment, commonly adopted for carbon fibers [13], does not represent a feasible solution, since an increase of the temperature above 300 °C leads to

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a severe weakening of fibers, attributed to a change in the crystalline structure of the fibers [15].

The improvement of the adhesion properties between basalt fibers and thermoplastic matrix is often based on indirect measurements, including impact resistance, yield strength, or short beam shear strength [16]. On the other hand, it is well known that direct measurements, such as single fiber fragmentation tests (SFFT), provide a direct measurement of the interfacial shear strength (IFSS) between matrix and reinforcement [13,17].

Therefore, the aim of this paper is the analysis of the effect of different treatments on the mechanical properties of basalt fibers, and the adhesion properties to polypropylene matrix. In particular, different sizing of the fibers, and the modification of matrix by the use of maleic anhydride grafted PP, have been studied. Mechanical properties of basalt fibers were studied by single filament tensile tests. The adhesion to PP matrix was studied by single fiber fragmentation tests (SFFT).

2. Materials and methods

2.1. Materials

Different types of basalt fibers were used:

(1) Commercial fibers Basaltex KVT150tex13-I, surface modified with a silane sizing and characterized by a nominal diameter of 13 μm, tensile modulus of 85 GPa and strength of 1.88 GPa, according to the technical data sheet, labeled as CB1. The average diameter of these fibers, measured by optical microscopy, is 14.5 + 1.56 μm.
(2) Commercial fibers Sudaglass BCR-3090-13, modified with a silane sizing, and characterized by a nominal diameter of 13 μm, tensile modulus of 85 GPa and strength of 2.15 GPa, according to the technical data sheet, labeled as CB3. The average diameter of these fibers, measured by optical microscopy, is 13.3 ± 0.48 μm, comparable to the value measured for fibers CB1.
(3) Prototype fibers OB1, obtained by a melt spinning process in Termotech plant, unsized, characterized by an average diameter of 16.01 ± 1.1 μm.
(4) Prototype fibers OB2, treated with a proprietary process including a polypropylene based wax, characterized by an average diameter of 12.6 ± 0.7 μm.

For comparison purposes, also glass fibers (Gun Roving E-Glass 2400 Tex by Fiberchina) were tested.

The matrix used is a polypropylene PP homopolymer, Moplen HP456J by Basell, extrusion grade, labeled as PPu. The matrix was modified by addition of 5% Licomont AR 504 (by Clariant) maleic anhydride grafted polypropylene wax. The modified matrix is labeled as PPm. The matrices were extruded and calendered using a Haake R Rheomex PTW 16/25 D twin screw extruder. The extrusion process was run at a screw temperature profile of 180–190–210–210–200 °C with a screw speed of 20 rpm. Films 0.2 mm thick were obtained by calendering.

Single filament tensile tests were performed according to ASTM 3379 using a TA Instruments ARES rheometer, operating in tensile mode. A single filament was bonded on a cardboard, which was then clamped between tools. 30 tests for each sample were performed, at a constant crosshead rate of 0.002 mm/min on lengths of 10, 20, and 40 mm.

Samples for single fiber fragmentation test (SFFT) were prepared by compression molding under a 20 MPa pressure on a single filament placed between two matrix films [18]. Non isothermal compression molding was performed using steel tools at room temperature, after preheating the matrix and fiber at 180 °C. SFFT tests were performed on a Lloyd LR 5K dynamometer, equipped with tensile tools, a load cell of 100 N and using a crosshead speed of 0.05 mm/min. The length of the broken filament segments, as well as the diameter of the filaments, was measured by using a Zeiss Imager A2M microscope.

X ray analysis was performed on basalt fibers within the range $2\theta = 10–40°$, using a Wide angle X-ray Diffractometer, RIGAKU Ultima+.

A Scanning Electron Microscope Zeiss EVO 40 SEM instrument was used for the morphological characterization of the surface of basalt fibers.

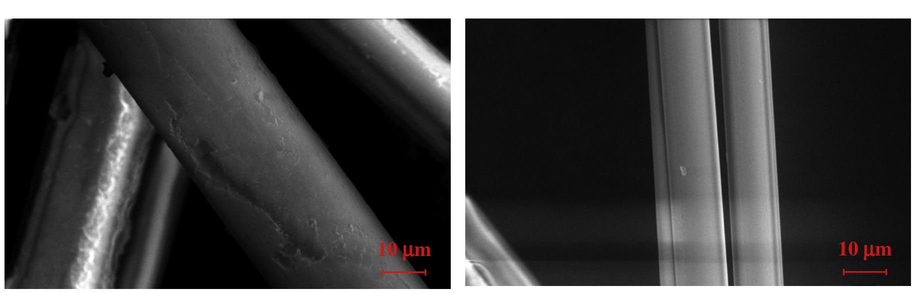
3. Results and discussion

Initially, SEM analysis performed on basalt fibers highlights the relevance of sizing on the surface microstructure of basalt fibers. In facts, unsized prototypal fibers OB1 are characterized by a rough surface, and the presence of significant surface defects as highlighted in Fig. 1(a). In contrast, sized basalt fibers OB2 are characterized by a very smooth surface, as evidenced by the micrograph reported in Fig. 1(b). Other commercial fibers, CB1 and CB3, are characterized by a surface morphology similar to that of fibers OB2.

Typical stress–strain curves for fibers CB1 measured at different gauge length L_F are reported in Fig. 2.

The tensile modulus of the basalt fibers measured at different length is reported in Fig. 3(a). The tensile strength is reported in Fig. 3(b). For comparison purposes, also the modulus and strength of glass fibers is reported in Fig. 3(a and b).

The mechanical properties of the filaments can be dependent either on the type of the fiber, or on the length of the filament. Therefore, two-way ANOVA was performed in order to highlight the effect of the two factors on the tensile modulus and tensile strength of filaments [19]. Initially, the basic requirement of ANOVA of comparable standard deviations was verified by Levene test [20]. The test statistics L was calculated as:



(a) OB1 fibers (b) OB2 fibers

Fig. 1. SEM micrographs of prototypal basalt fibers.