Composites Part B 145 (2018) 121–128

Contents lists available at ScienceDirect

Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

Structure-property relationships of basalt fibers for high performance applications

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ARTICLE INFO

Keywords:
Basalt fiber
Defects
Mechanical properties
Statistical properties/methods

ABSTRACT

Continuous basalt fibers present a unique combination of good mechanical and functional properties at an attractive cost level. These characteristics can be useful in high performance polymer matrix composites where cost orientation and function integration are becoming priority objectives. The present work examines a currently unresolved problem of basalt fibers regarding high performance applications: the variation of fiber structural and mechanical properties due to inhomogeneity of the natural raw material and the spinning process. Commercial basalt fibers from seven different manufacturers were investigated regarding their fiber chemistry, diameter distribution and occurrence of defects and crystallites. In a second step, structure-property relations on different scales were established with single fiber, impregnated roving and composite tensile test results.

1. Introduction

In recent years, continuous basalt fibers (CBF) arouse more and more interest from the scientific and industrial composites community [1,2]. Modulus and strength values for high grade basalt fibers lie right between E- and S2-glass fibers. With a superior performance to cost ratio compared to the common glass fibers, basalt fibers address the increasing cost-orientation across the industries currently using FRP. The thermal, chemical and tribological resistance and heat insulation properties of basalt fibers can represent an added value in function integrated composite structures.

For the application of basalt fibers in high performance industries such as aeronautics, the expectable scatter in CBF's structural and mechanical properties is seen as critical. Variations can originate from the utilized natural raw material and complications during the basalt fiber spinning process [3]. Prior to the use of CBF in high performance applications, this aspect requires further examination. Reproducible, measurable and constant material properties are a prerequisite for lightweight and safe design.

The selection of eligible basaltic rock as a raw material determines the chemical fiber composition which resembles synthetic glass fibers in the main oxides. The melt spinning process, currently used for the manufacturing of CBF, requires above 46% silica (SiO_2) [4]. Higher percentages further improve the spinning process [5] and enhance mechanical fiber properties. Likewise, alumina (Al_2O_3) improves mechanics depending on its quantity [6]. A main distinction between basalt and glass fibers is the high percentage of iron. The amount and share of ferric (Fe_2O_3) and ferrous (FeO) iron affect thermal and crystallization properties as well as the mechanical and optical characteristics of CBF [7–11]. Additional oxides that are usually present in CBF are CaO, MgO, Na_2O, K_2O and TiO_2.

The steps of the CBF spinning process are described by several authors in the literature [3,12]. At a given melt chemistry, pressure and drawing speed, high-grade filaments can only be spun at a certain temperature process window. Noncompliance can result in melt dripping, fiber breaks, defects and poor mechanical properties. Basalt fibers have a narrower processing window than glass and a higher variation of processing conditions throughout the nozzles of the bushing. In Fig. 1, the temperature process window (horizontal limits) and variation of process parameters (vertical error bars) are schematically compared between E-glass and basalt fibers. The processing window of stable fiber formation from vitreous melts is largely determined by the ratio of viscosity and surface tension [11,12]. High contents of silica, alumina and iron in the basalt rock cause an elevated viscosity and high spinning temperature [6,10,13]. Furthermore, the strong temperature dependency of the viscosity narrows the temperature window [14]. Additional confinement at high temperatures is caused by high wetting of basalt melts on the platinum-rhodium bushing [15]. Due to the decreasing contact angle, the melt can spread on the bushing surface instead of forming a stable spinning cone. As a further effect,

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https://doi.org/10.1016/j.compositesb.2018.03.028
Received 12 November 2017; Received in revised form 16 February 2018; Accepted 13 March 2018
Available online 15 March 2018
1359-8368/

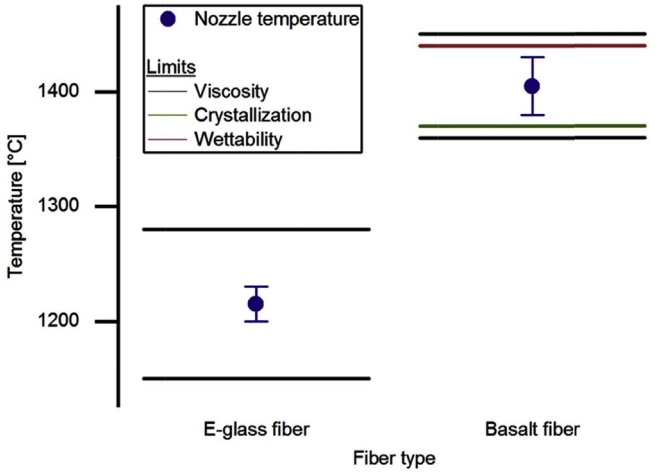

Fig. 1. Limits and homogeneity of basalt and E-glass fiber spinning. Values for the temperature windows are adopted from Ivanitskii [14].

crystallization can occur at the lower temperature limit but also during uncontrolled cooling of the spun basalt fibers [5]. Crystallites can act as defects in the fibers and reduce the mechanical strength. Compared to glass fibers, the CBF processing conditions throughout the nozzles of a bushing are subject to higher variation. This is mainly due to thermal insulation of basalt melt and the blockage of thermal radiation caused by its infrared absorbing characteristics. The resulting thermal inhomogeneity in the melt and on the bushing causes variation of viscosity, surface tension and other temperature-dependent spinning parameters.

It can be concluded that basalt rock has a more complex and less defined chemical composition than the industrially batched raw material of conventional glass fibers. Furthermore, the spinning of basalt fibers must occur within a comparatively narrow temperature window and is prone to variation of process parameters. On the other hand, high performance structural applications require a stable material and process chain. Critical variation in the fiber quality must be detectable and its influence on the composite properties has to be well understood.

The aim of this investigation is to quantify material and process induced variations in CBFs and to evaluate their impact on high-performance applications with the following approach:

- Structural and mechanical characterization of a set of commercially available basalt fibers, reference aerospace-grade E-, R- and S2-glass fibers.
- Analysis of the fiber's structural properties in terms of fiber defects, crystallinity, variation of chemical composition and the spinning process.
- Evaluation of the effect and importance of process and raw material induced variances on the fiber, impregnated roving and composite's mechanical properties (structure-property relationships).

2. Experimental

2.1. Materials

Off the shelf continuous basalt fibers from seven manufacturers were investigated. Fiber samples were sourced as roving with target specifications as follows: fineness of 600 tex, a mean diameter of 9 µm and epoxy compatible sizing based upon silane. The basalt roving specimens are labeled CBF1 to CBF7 to distinguish between the seven manufacturers. From three manufacturers (CBF1, CBF4 and CBF5), roving was sourced from three different production dates with a time offset of more than one week. These different spinning batches are indicated via an extra digit (e.g. CBF4.2). Aerospace grade E-, R- and S2-glass fibers, denoted by EGF, RGF and S2GF respectively were included as reference fibers. HexFlow® RTM 6 epoxy resin was used for the manufacturing of composite laminates and embedded roving specimens.

2.2. Analysis and test methods

2.2.1. Chemical composition

Chemical fiber compositions in mass and mole fractions of oxides were calculated from the results of a sequential energy-dispersive X-ray spectroscopy (EDX) and Moessbauer spectroscopy. Chemical composition data were evaluated regarding variation (e.g. between material batches) by calculating the pooled standard deviation s_p according to equation (1) [16]. At first, the standard deviation s_i is calculated in atomic percent for each of the nine constituents i of the compared compositions. Secondly, s_p is determined by weighting s_i with the respective average percentage n_i of the composition.

$$s_p^2 = \frac{\sum_{i=1}^{k} (n_i - 1) s_i^2}{\sum_{i=1}^{k} (n_i - 1)} \quad (1)$$

2.2.2. Diameter variation

For the determination of fiber diameter distributions, roving sections were impregnated with resin of a high resin-fiber contrast. After embedding, the specimens were cut, grinded and polished in a plane perpendicular to the fiber orientation. The profile of the entire roving was recorded with a Carl Zeiss AX 10 optical microscope by using a resolution of nine pixels per µm^2. The contrast threshold function of the ImageJ software was applied to the picture in order to distinguish fiber and matrix areas. The equivalent diameter for each fiber could be calculated from the related pixel areas.

The lengthwise deviation of fiber diameters was determined in a VEGA II TESCAN scanning electron microscope (SEM). Filament sections of 200 mm were measured for their diameters in steps of 20 mm.

2.2.3. Crystallinity

X-ray diffraction (XRD) was performed in a Seifert XRD 3003 TT powder diffractometer using CuK_α radiation with $\lambda = 1.54$ Å. Fibers were grinded with pestle and mortar. The collection of XRD patterns occurred in an angular range of $2\theta = 10$–70° at a scan step $2\theta = 0.05^\circ$ and a scan rate of $3^\circ 2\theta$/min.

2.2.4. Defect density

The fiber defect density was investigated on the basis of single fiber strength data (see 2.2.6.). The scale parameter σ_0 and the local Weibull modulus m of a two-parameter Weibull distribution [17] were graphically determined in a Weibull diagram. Subsequently, the mean filament strength at each of the four gauge lengths is calculated. Basalt fibers commonly exhibit brittle and weakest link failure characteristics [18,19]. Testing at different gauge lengths varies the fiber volume and surface which could potentially be affected by defects. As a consequence, fibers with high defect probability show a strong decrease of strength at higher gauge lengths while a perfect fiber would maintain the strength value from low gauge lengths. To use this behavior for conclusions on the defect density, mean strength values are plotted against different gauge lengths. According to the weakest link theory, the data points form a straight line in a log-log diagram assuming unimodal defect distribution [20]. The mean Weibull modulus ρ was determined from the slope of the linear fit equating to $-1/\rho$ [21,22]. A high value of ρ indicates a minor decrease of strength at higher gauge length and a low defect density.

2.2.5. Fiber density and roving fineness

Data on fiber density are required for all mechanical test methods, including the vibroscopic diameter measurement and fiber volume