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The Effect of Thermal Treatment on Tensile Properties of Basalt Fibers

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Abstract

The use of basalt fibers as a reinforcement in various composites requires the application of elevated temperatures during synthesis and processing conditions depending on the technique utilized. In this study, the tensile strength of basalt fibers at room temperature and also after exposure to 300, 350, 400, 450 and 500 °C in a furnace for durations of 5, 10, 15 and 20 min was investigated. The results indicate that the residual strength of basalt fibers drastically decreases after 20 min exposure at 300 and 400 °C and is only about 57 % and 35 % of that of fibers at room temperature, respectively. At 450 and 500 °C, this drastic decrease occurs after only 5 min. These results indicate the optimum conditions for processing parameters for basalt fibers and the composites based on them.

Keywords: Basalt fibers, monofilament, bundle, tensile and residual strength.

I. Introduction

Several studies have shown that fiber-reinforced composites are more efficient than other types of composites. The most common continuous fibers used for metal matrices are boron, graphite (carbon), alumina, and silicon carbide. These ceramic fibers are used in a variety of industrial and commercial applications. Although these fibers offer superior properties such as high-temperature stability, low thermal conductivity, low heat storage, their production cost is high. On the other hand, basalt fiber has been used as an inexpensive reinforcing composite material for the construction industry (e.g. asphalt, concrete) and also for making polymer matrix composites [1,2].

The manufacture of basalt fibers on industrial scale dates back to 1972, mainly for military and aerospace applications. It was only after 1995 that they began to be widely used in non-military applications [3].

The raw material for production of basalt fibers is basalt rock, a hard volcanic stone rich in Mg and Fe silicates found in both amorphous and crystalline states. Basalt rock is the most abundant volcanic stone in the earth's crust, covering areas of thousands of square kilometers and usually seen in black, dark brown and green colors [4].

The chemical composition of basalt includes: 42 – 60 % SiO_2, 13 – 18 % Al_2O_3, 8 – 15 % Fe_2O_3, 3 – 9 % MgO, 6 – 12 % CaO, 2 – 6 % Na_2O, 1 – 3 % TiO_2, 0.5 – 2 % K_2O and a small amount of Mn_2O_3. This wide-ranging chemical composition is mainly due to different environmental conditions during the formation of the rocks in nature [5–7].

Basalt fibers are manufactured by means of crushing and melting of basalt rock at 1500 °C. This melt is then transferred onto a rotating disk, which contains several tiny holes (bushings) and then drawn through the bushings by means of air blowing. The produced fibers are subsequently covered with a polymer sizing that improves the corrosive and wear resistance of the fibers [8–10]. According to the literature, the sizing content for basalt and E-glass fibers is about 0.1 to 0.2 wt% [11].

Compared to E-glass, basalt fibers exhibit superior mechanical properties, higher chemical stability and better thermal/electrical insulation, which makes these fibers an attractive reinforcement for polymer matrix composites. These fibers are also used for reinforcement of concrete structures, asphalt and a number of plastics [12–15]. In more recent studies, attempts have been made to use basalt fibers as reinforcement for metal matrix composites [16–20].

The thermal properties of basalt products have been the subject of several investigations [21,22]. In this study, the effects of temperature exposure and also the duration of heating on the tensile properties of basalt fibers have been investigated. This is particularly significant to select the manufacturing method for basalt-reinforced composites and/or the subsequent thermal treatment of composites based on them. The thermal stability of basalt fibers placed in a furnace and exposed to different temperatures was evaluated. The factors considered for exposure were time and temperature.

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II. Experimental Method

(1) Basalt fibers

In this study continuous basalt roving from Ukraine was used as the raw material. The average diameter of a single fiber measured by CamScan MV2300 Scanning Electron Microscope (SEM) was approximately 10 mm (Fig. 1). The density of fibers quantified based on the Archimedes principle using an electronic balance with an accuracy of 0.1 mg was 2.816 g/cm^3. Hence, the number of fibers in the bundle was calculated to be about 1124.

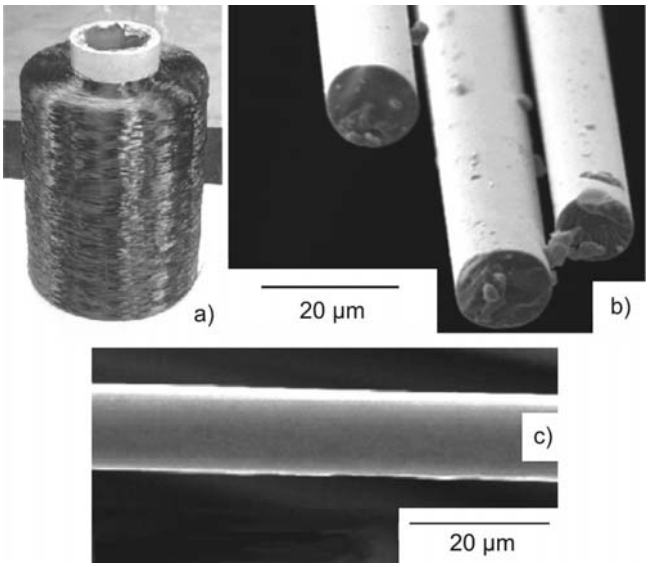

Fig. 1: A roving of basalt fibers (a) and scanning electron microscope (SEM) image of the monofilaments (b, c).

(2) Tensile strength

Tensile strength testing was done on single fibers in accordance with the modified ISO 11566 standard [23]. The effect of temperature exposure on the tensile behavior of basalt fibers was studied by heating fibers in a furnace at different temperatures (300, 400, 450 and 500 °C) for predetermined times (5, 10, 15 and 20 min) and subsequently cooling in room temperature. The tensile strength of basalt fibers was measured and the percentage of residual strength was calculated in relation to the initial room temperature strength of the bundles.

The test apparatus consisted of an Instron 5565 tensile tester equipped with a 2.5 N load cell and a crosshead speed of 2 mm/min. The gauge length was kept at 25 mm.

III. Results

(1) Tensile strength at room temperature

In order to find the range of processing parameters for basalt fibers, it is essential to measure the residual strength of basalt fibers at elevated temperatures, calculated in relation to their room temperature strength.

The tensile strength of basalt monofilament and fiber bundles at room temperature was measured ten times and then averaged to about 1 846 and 322 MPa, respectively. This significant difference is mainly due to the accumulation of defects on the bundles as compared to a monofilament. As estimated, each bundle consists of about 1 124 monofilaments and therefore the number of defects in a certain length of a bundle compared to that of a single filament increases drastically. After rupture of a defective monofilament, the load is applied on fewer fibers, which results in more stress concentration and rupture of the remaining fibers.

(2) Tensile strength after thermal treatment

The purpose of this section is to study the potential applicability of basalt fibers as the reinforcement of composites at elevated temperatures for production or post-production stages (e.g. heat treatment, curing, thermal bonding), in order to make a decision on the range of processing parameters. The average strength of basalt bundles after exposure to different temperatures for different times and their residual strength are presented in Tables 1 and 2.

Table 1: Tensile strength (MPa) of basalt bundles for various temperatures and holding times.

T (°C)	Holding Time (min)			
	5	10	15	20
300	255	250	248	189
400	193	177	171	113
450	76	72	61	49
500	73	67	57	36

Table 2: Residual strength (%) of basalt bundles for various temperatures and holding times.

T (°C)	Holding Time (min)			
	5	10	15	20
300	79	78	77	58
400	60	55	53	35
450	24	22	19	15
500	23	21	18	11

The variation of residual strength with holding time for different temperatures is also shown in Fig. 2. As can be seen, about 20 % of the strength of fibers is lost during the first 5 min of holding at 300 °C. The increased holding time up to 15 min does not appear to have any significant effect on the strength, but this value decreases drastically within 15 to 20 min of exposure and only 58 % of the room temperature strength has remained.

The same trend is observed for fibers exposed to 400 °C with a reduced strength during the whole period of time as compared with 300 °C. As can be seen from Table 2, only 24 % of the initial room temperature strength of the basalt fibers remains after 5 min exposure to 450 °C and decreases gradually to 15 % after 20 min. The increased temperature up to 500 °C imposed a slight decrease in the fiber strength for the whole period of exposure time.

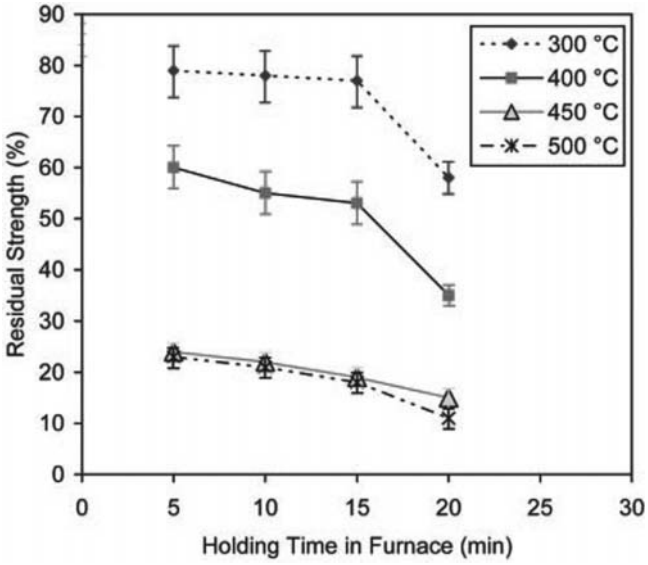

Fig. 2: The residual strength of fibers vs. holding time at different temperatures.

The results reveal that further increase of the exposure time (e.g. up to 20 min) even at increased temperatures (e.g. 500 °C) did not show any significant effect on the residual strength of the fibers. These results indicate that when these fibers are used as reinforcements, the maximum permitted temperatures and exposure times during processing or service are 300 – 400 °C and 15 min, respectively.

(3) Degradation mechanism of basalt fibers

Based on the literature, the main factor determining the heat temperature stability of basalt fibers is their crystallization behavior, which depends on chemical composition and heat treatment conditions [24]. The degradation mechanism of basalt fibers at elevated temperature may be explained based on the Weibull model [25], and due to nonehomogeneities, which behave as defects along the fiber volume. Basalt fibers are initially in amorphous state, but can gradually crystallize in localized areas at elevated temperatures. These transformed crystallites are more brittle and result in lower strength values [22, 24, 26].

The elevation of temperature increases the number of defects exponentially, leading to a drastic decrease in tensile strength. The fracture of fibers takes place as a result of a distortion between amorphous and localized crystalline areas, which may be present in the fiber structure [27].

Militky *et al.* also studied the effect of thermal treatment on the tensile failure of basalt and glass fibers [22], based on a statistical model proposed by Wagner [25]. They reported that treatment of basalt fibers only above 300 °C led to a significant drop in strength and structural changes, which is in agreement with the results of this study.

The microscopic studies of the work [22] and the ones obtained in this study confirm more localized fracture zones on the fibers treated at higher temperatures, as compared to the ones at ambient temperatures. Fig. 3 shows the SEM fracture plane of an untreated basalt fiber in this study as compared to that obtained in work [22]. Fig. 4 shows the SEM images of fracture surfaces of basalt bundles after exposure at 400 °C for 10 min with different magnifications. As can be seen, fracture has occurred in different planes, suggesting that defects have formed at various levels.

A secondary phenomenon, reported in the literature, affecting the mechanical properties of basalt fibers is the sizing burn-off at elevated temperatures, and therefore exposure of bare fibers to the atmospheric environment at elevated temperatures, which could cause corrosion and formation of local defects contributing to failure of fibers. Manylov *et al.* [27] reported that heat treatment of basalt fibers in air leads to iron oxidation and the bulk growth of particles, which act as nucleation sites for pyroxene.

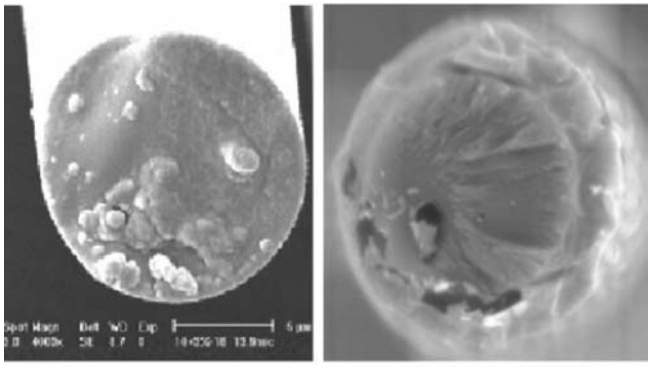

Fig. 3: SEM micrographs of fractured planes of a basalt monofilaments: a) obtained in this study, b) from work [22] (magnification 10 000 times).

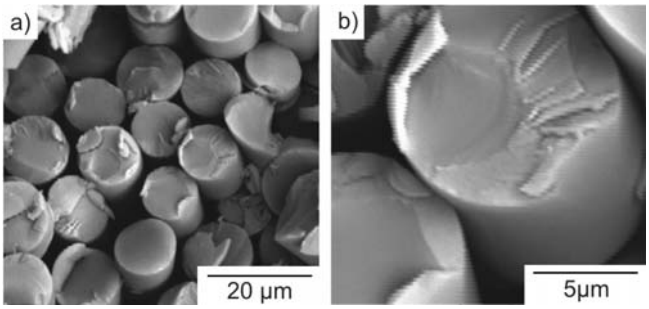

Fig. 4: SEM micrographs of fractured basalt bundles after thermal treatment at 400 °C for 10 min with different magnifications.

The temperature-induced mechanism of strength degradation at relatively low temperatures is a phenomenon reported by others, as well. Militky *et al.* [22] reported crystallization of basalt fibers at temperatures above 700 °C, which is in accordance with data given by Makhova [28] earlier, while Karamanov *et al.* [29] report temperatures > 600 °C for crystallization of these fibers. As the data in the literature is not straightforward and not in accordance with each other, additional experimental studies (e.g. TEM microscopy) on heat-treated fibers seem to be mandatory for a detailed interpretation of the fracture mechanisms of these fibers, which was not possible in the time framework of this study.

IV. Conclusions and Future Work

The basalt fibers used in this study had the density of 2.816 g/cm^3 and an average diameter of 10 µm. The strength of a single filament was about 1 846 MPa and that of a bundle was around 322 MPa. The strength of these fibers deteriorates gradually with time when subject

to relatively high temperatures. This effect, which is attributed to an amorphous-to-crystalline transformation, is accelerated after 15 min exposure at 300 or 400 °C and is maximized at 450 °C. Based on the results, the maximum permitted temperatures and exposure times during processing and service conditions of basalt fibers are 300 – 400 °C and 15 min, respectively.

Other researchers have also reported the temperature-induced mechanism of strength degradation of basalt fibers at relatively low temperatures. Based on the literature, it appears that degradation of basalt fibers is mainly due to distortion between amorphous and localized crystalline areas and also the sizing burn-off, which exposes the intact fibers to the atmospheric environment and possible corrosion and oxidation of iron on the fibers.

Since the data interpretation of the literature in regards to crystallization of basalt fibers is not straightforward, additional experimental studies on heat-treated basalt fibers are mandatory, which was not possible in the case of this study. TEM microscopy of the fracture surface of the fibers seems to be ideal for the study of early crystallization phenomenon. Alternatively, XRD, DTA or DSC studies may give more indications with regard to the formation of newly crystalline locals at different temperatures.

References

1 Goldworthy, W.B.: New basalt fiber increases composite potential, *Compos. Technol.*, **8**, 1 – 15, (2000).

2 Singha, K.: A short review on basalt fiber, *International Journal of Textile Science*, **1**, [4], 19 – 28, (2012).

3 Kostikov, V.I., Makhova, M.F., Sergeev, V.P., Trefilov, V.I.: Ceramic fibers, in *Fibre Science and Technology (Soviet Advanced Composites Technology Series)*, J. N. Fridlyander and I. H. Marsball, Eds. Springer, 1995, 580 – 603.

4 Czigány, T.: Special manufacturing and characteristics of basalt fiber reinforced hybrid polypropylene composites: mechanical properties and acoustic emission study, *Compos. Sci. Technol.*, **66**, [16], 3210 – 3220, (2006).

5 Rapp, C.F., Fausey, W.H., Gonterman, J.R.: Basalt compositions and their fibers, US Patent No. 4560606, 1985.

6 Schiavon, M.A., Redondo, S.U.A., Yoshida, I.V.P.: Thermal and morphological characterization of continuous basalt fibres (in Portuguese), *Cerâmica*, **53**, 212 – 217, (2007).

7 Parnas, R., Shaw, M.: Basalt fiber reinforced polymer composites – prepared for the new england transportation consortium, (2007).

8 Weddell, J.K.: Continuous ceramic fibres, *J. Text. Inst.*, **81**, 333 – 359, (1990).

9 Brik, V.B.: Multifunctional apparatus for manufacturing mineral basalt fibers, US Patent 6647747, (2003).

10 Kamiya, S., Sasaki, H. Nakagawa, N.: Method for manufacturing basalt fiber, US Patent 8501643, (2013).

11 Wei, B., Cao, H., Song, S.: Environmental resistance and mechanical performance of basalt and glass fibers, *Mater. Sci. Eng. A*, **527**, 18 – 19, 4708 – 4715, (2010).

12 Sim, J., Park, C., Mon, D.Y.: Characteristics of basalt fiber as a strengthening material for concrete structures, *Compos. Part B Eng.*, **36**, [6 – 7], 504 – 512, (2005).

13 Amuthakkannan, P., Manikandan, V., Jappes, J.T.W., Uthayakumar, M.: Effect of fibre length and fibre content on mechanical properties of short basalt fibre reinforced polymer matrix composites, *Mater. Phys. Mech.*, **16**, 107 – 117, (2013).

14 Van de Velde, K., Kiekens, P., Van Langenhove, L.: Basalt fibers as reinforcement for composites, Zwijnaarde, Belgium.

15 Ibrahim, A., Faisal, S., Jamil, N.: Use of basalt in asphalt concrete mixes, *Constr. Build. Mater.*, 23, 498 – 506, (2009).

16 Sabet, S.M.M., Akhlaghi, F., Eslami-farsani, R.: Production and optimization of aluminum-basalt composites by hand layout technique, in *Proceedings of the World Congress on Engineering*, **Vol. III**, 1826 – 1829, (2012) .

17 Akhlaghi, F., Eslami-Farsani, R.,Sabet, S.: Synthesis and characteristics of continuous basalt fiber-reinforced aluminum matrix composites, *J. Compos. Mater.*, **47**, [27], 3379 – 3388, (2012).

18 Khalili, S.M.R., Daghigh, V., Farsani, R.E.: Mechanical behavior of basalt fiber-reinforced and basalt fiber metal laminate composites under tensile and bending loads, *J. Reinf. Plast. Compos.*, **30**, [8], 647 – 659, (2011).

19 Antonov, M., Surženkov, A., Hussainova, I., Goljandin, D., Mikli, V.: Effect of basalt addition on tribological performance of FeCrSiB HVOF coatings, *Est. J. Eng.*, **18**, [3], 211 – 220, (2012).

20 Hrynyk, R., Frydrych, I., Irzmanska, E., Stefko, A.: Thermal properties of aluminized and non-aluminized basalt fabrics, *Text. Res. J.*, **83**, [17], 1860 – 1872, (2013).

21 Rambo, D.A.S., de Andrade Silva, F., Toledo Filho, R.D., da Fonseca Martins Gomes, O.: Effect of elevated temperatures on the mechanical behavior of basalt textile reinforced refractory concrete, *Mater. Des.*, **65**, 24 – 33, (2015).

22 Militky, J.V., Kovacic, V., Rubnerova, J.: Influence of thermal treatment on tensile failure of basalt fibers, *Eng. Fract. Mech.*, **69**, [9], 1025 – 1033, (2002).

23 Carbon fiber-determination of the tensile properties of single-filament specimens, ISO 11566, (1988).

24 Fiore, V., Scalici, T., Di Bella, G., Valenza, A.: A review on basalt fibre and its composites, *Compos. Part B Eng.*, (2015).

25 Wagner, D.H.: Stochastic concepts in the study of size effects in the mechanical strength of highly oriented polymeric materials, *J. Polym. Sci. Part B Polym. Phys.*, **27**, [1], 115 – 149, (1989).

26 Rubernova, J.: Thermomechanical properties of inorganic fibers, Technical University of Liberec, 1996.

27 Manylov, M., Gutnikov, S., Pokholok, K., Lazoryak, B., Lipatov, Y.: Crystallization mechanism of basalt glass fibers in air, *Mendeleev Commun.*, **23**, [6], 361 – 363, (2013).

28 Makhova, M.F.: Crystallization of basalt fibers, *Glas. Ceram.*, **25**, [11], 672 – 674, (1968).

29 Karamanov, A., Pelino, M.: Crystallization phenomena in iron-rich glasses, *J. Non. Cryst. Solids*, **281**, [1 – 3], 139 – 151, (2001).