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Ultimate Mechanical Properties of Thermally Exposed Basalt Filament Yarns

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Fresh basalt fibers are practically amorphous. Due to high temperature action these fibers have ability to partially crystallize. This form of basalt fibers can be more brittle and their strength can be too low. In this contribution the strength of basalt filament yarns is investigated at room temperature and after tempering to the **50, 100, 200, 300, 400** and **500°C.** Structural changes of fibers are identified by scanning electron microscopy. The strength drop of basalt filaments after long term temperature exposition is described by the linear spline nonparametric model.

Keywords: basalt fibers; ultimate strength; tempering influence

1. INTRODUCTION

Basalt fibers as well as glass fibers can be used for production of high temperature resistant and chemically inactive products. Main problems of basalt fibers preparation are due to gradual crystallization of some structural parts (plagioclase, magnetite, pyroxene) and due to non-homogeneity of melt. Basalt is therefore still used mainly for molded products (flag stones, pipes) with increased abrasion resistance, temperature resistance and chemical resistance. Basalt is also used in a form of short fibers for insulation purposes (basalt wool). Basalt yarns are still used only rarely.

Utilization of the technology of continuous spinning overcomes the problems with unevenness and final filament yarns are applicable in the textile branch. It is possible to use these yarns for production of planar or 3D textile structures for composites, special knitted fabrics and also as the sewing threads. Especially an application of basalt yarns as the sewing threads is very

attractive. It is possible to use these threads for joining of filtering bags for hot media, filtering bags for very aggressive chemical environment, etc.
The fresh basalt fibers are practically amorphous. Due to high temperature action these fibers have ability to partially crystallize. This modified form of basalt fibers can be more brittle and their strength can be too low.

In this contribution the changes of selected properties of basalt fibers after thermal exposition are presented. These properties are investigated at room temperature and after tempering to the **50, 100, 200, 300, 400** and **500°C**. The ultimate strength, deformation at break and sound wave spread velocity are measured.

2. BASALT FIBERS

Basalt is a generic name for solidified lava which poured out of the volcanoes [1, 2, 5, 6]. Basaltoid rocks are melted approximately within the range 1500 - 1700°C. When the melt is quickly quenched, it solidificates to glass-like amorphous solid. Slow cooling leads to more or less complete crystallization, to an assembly of minerals. Two essential minerals plagiocene and pyroxene make up perhaps 80% of lots of basalts. Classification of basaltoid rocks based on the contents of main basic minerals is described in the book [5].

Basaltoid rocks, which are suitable for creation of fibers, contain in most cases two minerals:

Olivine ($2(MgFe).O.SiO_2$)
Nepheline ($Na_2O.Al_2O_3.2SiO_2$).

From the point of view of basalts chemical composition the silica oxide SiO_2 (optimal range 43.3 - 47%) dominates and Al_2O_3 (optimal range 11 - 13%)is next in the abundance. Content of CaO (optimal range 10 - 12%) and MgO (optimal range 8 - 11%) is nearly similar Other oxides are almost always

below 5% level. According to the SiO_2 content basalt rocks are divided into the three main groups:

Alkaline basalt	up to 42% of SiO_2
Mildly acid basalt	from 43% to the 46% of SiO_2
Acid basalt	over 46% of SiO_2

Basalt color vary from brown to dully green in dependence on the ferrous oxides content.

Basalts are more stable in strong alkalis that glass. Stability in strong acids is slightly lower. Basalt products can be used from very low temperatures (about -200° C) up to the comparatively high temperatures 700 - 800° C. At higher temperatures the structural changes occur.

Basalt rocks for the fibers preparation have to follow these requirements:

(i) SiO_2content over 46% (acid type) with constant composition

(ii) ability to melt without solid rests

(iii) optimal melt viscosity for fibers formation

(iv) ability to solidificate into the glassy state (without marked crystallinity)

In the manufacturing of fibers, the basic technological criterion is provided by the acidity coefficient

$$M_k = (SiO_2 + Al_2O_3) / (CaO + MgO)$$

Value of M_k should be in the range from 1.1 to 3.0. Ideal technological conditions for fiber creation are represented by the Mk = 1.65 [6]. More precise criteria which take into account the effect of individual oxides on viscosity of melt are given in [6].

In practice the suitability of basaltoid rocks for fibers preparation is based on their chemical and mineralogical composition. Attention should also be paid to the textural characteristic of the respective rocks [7].

Basalt rocks from VESTANY hill was used as a raw material in this work. Based on the DTA measurements the crystallization temperatures T_c of

individual minerals are evaluated. For Magnetite is T_c = 720 °C for Pyroxene T_c = 830 °C and for Plagioclase T_c = 1010 °C.

Basalt fibers as well as glass ones are prepared from melt (melting temperature is about 1500°C) on the same type of apparatus. Comparison of chemical composition of glass and basalt fibers is given in Table I.

TABLE I. Chemical Composition of Glass and Basalt Fibers (in weight %)

	E-glass	**S-glass**	**C-glass**	**Basalt**
SiO_2	52 - 56	65	64 - 68	51.56
Al_2O_3	12 - 16	25	3 - 5	18.24
CaO	16 - 25	-	11 - 15	5.15
MgO	0 - 5	10	2 - 4	1.3
B_2O_3	5 - 10	-	4 - 6	-
Na_2O	0.8	0.3	7 - 10	6.36
K_2O	0.8	0.3	7 - 10	4.5
TiO_2	-	-	-	1.23
Fe_2O_3	-	-	-	4.02
FeO	-	-	-	2.14
MnO	-	-	-	0.28
P_2O_5	-	-	-	0.26

Filament yarns contained 280 single filaments were used. Mean fineness of yarn was 45 tex.. The basic physical properties of basalt fibers are presented in Table II.

TABLE II. Basic Physical Properties of Glass and Basalt Fibers

Property	**E-glass**	**Basalt**
Diameter [μm]	9 - 13	8.63
Density [kgm^{-3}]	2540	2733
Softening temperature [°C]	840	960

3. STATISTICAL ANALYSIS OF FIBERS STRENGTH

The fracture of fibers can be generally described by the micromechanical models or on the base of pure probabilistic ideas [8]. The probabilistic approach is based on these assumptions:

(i) - fiber breaks at specific place with critical defect (catastrophic flaw),

(ii) - defects are distributed randomly along the length of fiber (model of Poisson marked process),

(iii) - fracture probabilities at individual places are mutually independent.

The cumulative probability of fracture $F(V, \sigma)$ depends on the tensile stress level and fiber volume V. The simple derivation of the stress at break distribution described for example by Kittl and Diaz [9] leads to the general form

$$F(V, \sigma) = 1 - \exp(- R(\sigma))$$

The $R(\sigma)$ is known as the specific risk function. For famous Weibull distribution has function $R(\ )$ the form [14]

$$R(\sigma) = [(\sigma - A)/B]$$

where A is lower strength limit, B is scale parameter and C is shape parameter (model WEI 3). For brittle materials it is often assumed A = 0 (model WEI 2).

The individual basalt filaments removed from yarn were tested. The loads at break were measured under standard conditions at sample length 10 mm. Load at break data were transformed to the stress at break σ_i [GPa]. The sample of 50 stress at break values was used for evaluation of the R(.) functions and estimation of their parameters.

Owing to their special structure the parameters of Weibull type distributions can be estimated by using of the maximum likelihood method. This method is very interesting because of its good statistical properties (asymptotic efficiency, consistency and asymptotic normality of estimators) [10].

In the case σ_i i=1,...N are independent random variables with the same probability density function $f(\sigma) = F'(\sigma_i, \mathbf{a})$ the logarithm of likelihood function has the form

$$\ln L = \Sigma \ln f((\sigma_i, \mathbf{a})$$

where **a** are parameters of corresponding risk function.

The MLE estimators $\mathbf{a}^*$ can be obtained by the maximization of the ln L(**a**). This task can be simply converted into solving of the set of nonlinear equations (see[10]). Estimates $\mathbf{a}^*$ obtained in such a way for three and two parameter Weibull distribution are given in Table III.

TABLE III. Parameters of Weibull models calculated by MLE

Model	A [GPa]	B [GPa]	C [-]	ln L(a^*)
WEI3	0.0641	0.230	1.370	33,50
WEI2	-	0.301	1.829	29.164

The SEM micrograph of typical broken basalt fiber (magnification 10 000) shows the occurrence of brittle fracture. The SEM of longitudinal portion of basalt fiber (magnification 10 000) shows that surface is very smooth without flaws or crazes. Based on these findings we can postulate that fracture occurs due to nonhomogenities in fiber volume (probably near the small crystallites of minerals).

5. THE PROPERTIES OF BASALT AFTER THERMAL EXPOSITION

Behavior of basalt filament yarns after long - term thermal exposition was simulated by tempering of fibers at the temperatures 50, 100, 200, 300°C. The time of exposition was 60 min. After tempering the following properties was measured:

- tensile strength [$N.tex^{-1}$]
- deformation at break [%]
- dynamic acoustic modulus [Pa] (dynamic acoustic modulus was determined from sound wave spread velocity in the material).

The changes of properties of basalt after tempering are investigated by the analysis of variance. It was determined that only 300°C tempering led to

the statistically significant drop of strength and dynamic acoustical modulus. Probably, the changes of these properties are based on the changes of the crystalline structure of fibers.

In the second set of experiments the strength distribution of basalt filament yarns was measured on the samples tempered in the oven at temperatures T_T = **20, 50, 100, 200, 300, 400** and **500°C** in selected time intervals t_T = **1, 15,**and **60 min**.

For strength evaluation the TIRATEST 2300 machine was used. The 50 samples of strength P_i are collected. These values were recalculated to stress at break values σ_i **[GPa].**

The strength distribution of tempered filament yarns was nearly Gaussian with parameters: mean value σ_p and variance σ^2.
These parameters are estimated by the sample arithmetic mean and sample variance. Results are given in the table IV.

TABLE IV .Parameters of Tempered Filament Yarn Strength

t_T [min]	1		15		60	
T_T[°C]	σ_p[GPa]	σ^2[GPa^2]	σ_p[GPa]	σ^2[GPa^2]	σ_p[GPa]	σ^2[GPa^2]
20	1.01	.0075	1.01	.0075	1.01	.0075
50	.997	.0110	1.05	.0110	1.07	.0150
100	1.03	.0095	.991	.0140	1.01	.0100
200	.986	.0091	1.01	.0083	1.09	.0110
300	.893	.0140	.743	.0150	.424	.0100
400	.743	.0061	.701	.0091	.112	.00150
500	.254	.0048	.348	.0026	.094	.00300

The dependence of the filament yarns strength on the temperature has two nearly linear regions. One at low temperature to the 180°C with nearly constant strength and one up to the 340°C with very fast strength drop.

For description of this dependence the linear spline model was used [11]. By the linear least squares the strength σ_1 for temperature T_1=180°C and σ_2 for temperature T_2 =340°C were computed. These values and the rate of strength drop

$$D = (s_1 - s_2)/ 160 \ [\text{GPa deg}^{-1}]$$

are given in table V.

TABLE V. Thermal Dependence of Filament Yarns Strength

t_T [min]	σ_1[GPa]	σ_2[GPa]	D[GPa deg^{-1}]
1	1.0074	.756	.0016
15	1.1070	.343	.0048
30	1.1750	.158	.0064

It is clear that increasing of the time of tempering leads to the acceleration of structural changes and drop of strength fastening (increasing D).

6. CONCLUSION

From thermal dependence of the filament yarns strength is evident that long term exposition at temperatures above 200°C leads to the drop of mechanical properties probably due to the gradual crystallization. This hypothesis was verified by the electron microscopy of fibers break zone.

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