Compressive Creep of Basalt Fibers and Epoxy Resin Linear Composite

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Basalt fibers similarly as a glass fibers are used for production of high temperature resistant and chemically inactive products. Basalt is used mainly for molded products (flag stones, pipes) with increased abrasion resistance, temperature resistance and chemical resistance or in a form of short fibers for insulation purposes (basalt wool).

Application of the technology of continuous spinning leads to the sufficiently even basalt filament yarns applicable in the textile branch. In this contribution the thermal expansion and compressive creep of basalt and composite with epoxy resin are investigated.

Keywords: Basalt fibers; compressive creep; longitudinal modulus; thermomechanical analysis

1. INTRODUCTION

Basalt fibers similarly as glass fibers can be used for production of high temperature resistant and chemically inactive products. The 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 used also in a form of short fibers for insulation purposes (basalt wool). Basalt filament yarns are therefore used only rarely.

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Utilization of the technology of continuous spinning overcomes the problems with unevenness and resulted 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 the strength can be too low.

In this contribution the compressive creep and thermal expansion of basalt and linear composite with epoxy resin are evaluated by using of the original thermomechanical analyzator TMA. The simple model foe estimation of basalt fibers longitudinal modulus is proposed.

2. BASALT FIBERS

Basalt is generic name for solidified lava which poured out the volcanoes [1, 2, 5]. Basaltoid rocks are melted approximately in the range $1500-1700°C$. When this melt is quickly quenched, it solidificated to glass like nearly 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 many 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 often green mineral Olivine ($2(MgFe).O.SiO_2$) and Nepheline ($Na_2O.Al_2O_3. 2SiO_2$).

From the point of view of chemical composition of basalts the silicon oxide SiO_2 (optimal range $43.3-47\%$) dominates and Al_2O_3 (optimal range $11-13\%$) is next in abundance. Content of CaO (optimal range $10-12\%$) and MgO (optimal range $8-11\%$) is closely similar Other oxides are almost always below 5% level. According to the SiO_2 content the basalt rocks are divided to 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

The color of basalt is from brown to the dully green in dependence on the ferrous oxides content.

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

Basalt rocks for the fibers preparation musts satisfy to the following requirements:

 (i) content over 46% of SiO_2 (acid type) with constant composition

 (ii) ability to melt without solid rests

(iii) optimal melt viscosity for fibers formation

(iv) ability to solidification to the glassy state (without marked crystallinity)

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

Basalt rocks from VESTANY hill was used as raw material in this work. Based on the DTA measurements the crystallization temperatures T_c of individual minerals are evaluated. For Magnetitie is $T_c = 720°C$ for Pyroxene $T_c = 830°C$ and for Plagioclase $T_c = 1010°C$.

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

The marbles, filament roving and linear composite with epoxy resin are prepared. From marbles the thick rods were prepared by grinding. The roving contained 280 single filaments are used. Mean fineness of roving was 45 tex. The linear composite rods containing 90% of basalt roving and 10% of epoxy resin (CHS 1200) were prepared. These rods were cut to the cylindrical samples having length 12.33 mm and diameter 5.137 mm. For comparative purpose the same forms of pure epoxy resins were prepared. The basic physical properties of basalt fibers are presented in Table II.

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

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. THERMOMECHANICAL ANALYSIS

For thermomechanical analysis the special device TMA CX 03RA was used. This apparatus enables to measurement of dimensional changes of material under specified compressive load in dependence on free programmable time temperature profile. For measurements the basalt rods (abbreviation R) and linear composite from roving glued by epoxy resin CHS 1200 abbreviation C) were used.

The dilatation curves *i.e.*, dependence of height of basalt rod on the temperature were measured at rate of heating 10 deg min^{-1} and compressive load 10 mN. These curves consists from two nearly linear portions connected at glass transition temperature T_g [3].

The coefficients of linear thermal expansions a for region below and above T_g were computed from models

$$L/L_g = 1 + \alpha_1^*(T_g - T) \text{ for } T \text{ below } T_g \qquad (1)$$

$$L/L_g = 1 + \alpha_2^*(T - T_g) \text{ for } T \text{ above } T_g \qquad (2)$$

By the nonlinear least squares the: $T_g = 596.3°C$, $\alpha_1 = 4.9 \, 10^{-6}$ deg^{-1}, $\alpha_2 = 19.1 \, 10^{-6} \, deg^{-1}$ were computed.

The response of basalt on the compressive loads under isothermal conditions were investigated from creep type experiments. The load was 200 mN. For the basalt rods and linear composites the dependence of sample height L on the time t were measured. The experimental data were described by the simple two exponential model [12]

$$L = L_p + L_1 \exp(-k_1^* t) + L_2 \exp(-k_2^* t) \qquad (3)$$

Typical result of nonlinear regression is shown on the Figure 1.

From parameters L_p, L_1, L_2, k_1 and k_2 estimated by the nonlinear least squares the maximum dilatation.

$$D = L_1 + L_2 \qquad (4)$$

and half time of dilatation $t_{1/2}$ were computed. Denote that $t_{1/2}$ is time for which is the dilatation equal to

$$L_p + (L_1 + L_2)/2. \qquad (5)$$
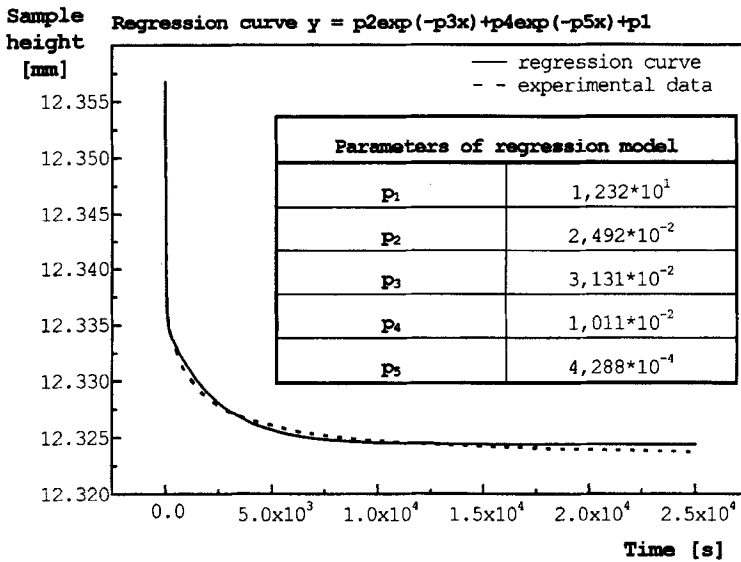
These parameters are given in the Table III.

FIGURE 1   Regression curve and experimental data for creep at 250°C.

TABLE III   Parameters of compressive creep

T[°]	Linear composite (C)		Basalt rod (R)	
	D[mm]	$t_{1/2}[s]$	D[mm]	$t_{1/2}[s]$
30	0.0030	145.1	0.0145	16015
50	0.0289	36.90	–	–
100	0.0532	29.80	0.0083	21.30
250	0.0350	41.10	–	–
300	–	–	0.0426	517

4. LONGITUDINAL COMPRESSIVE MODULUS OF BASALT FIBERS

Let one has the linear composite (index C) consisted from the phase of Basalt fibers (index K) and epoxy resin matrix (index E). Let the both phases are deformed elastically that their Poisson ratio is the same and that the stresses cause no debonding of the interfaces. The volumetric ratio of Basalt fibers (composite has the same length as individual phases) is Φ_K

From the simple rule of mixture (derivation is in [4]) it follows

$$E_C = \Phi_K E_K + (1 - \Phi_K)E_E \qquad (5)$$

Here E_C is longitudinal creep modulus of linear composite E_E is longitudinal creep modulus of epoxy resin and E_K is longitudinal creep modulus of basalt fibers. For known Φ_K and E_C, E_E the longitudinal compressive modulus of Basalt fibers is equal to

$$E_K = \frac{E_C - (1 - \Phi_K)E_E}{\Phi_K} \qquad (6)$$

For our case the Φ_K was estimated by the image analysis and value $\Phi_K = 0,9$ was obtained.

The modulus E_C at individual temperatures was computed as the ratio

$$E_C = \frac{F}{A_C \varepsilon_{E(30)}} \qquad (7)$$

where $F = 200\,mN$ is applied load, $A_K = 20.725\,mm^2$ is cross sectional area of tested composite sample and $\varepsilon_{K(10)}$ is deformation under

TABLE IV Longitudinal compressive modulus of linear composite, epoxy resin and basalt fibers

Temperature [°C]	E_K (Basalt) [GPa]	E_C (Composite) [GPa]	E_E (Resin) [GPa]
25	112.27	105.41	40.5129
50	95.256	87.49	17.5076
100	114.084	108.575	58.9869
200	99.76	90.95	11.6239

compressive creep in time $t = 10$ s. Values $\varepsilon_{K(10)}$ were computed from dimensional changes after 10 sec. of compressive creep for linear composite at individual temperatures.

By the same way the modulus E_E was estimated from the compressive creep curve of pure epoxy resin. In this case $F = 200$ mN, $A_E = 20.725$ mm^2 and $\varepsilon_{E(10)}$ were computed from dimensional changes after 10 sec. of compressive creep for epoxy resin at individual temperatures.

Computed values of E_E, E_C and E_K are summarized in the Table IV.

5. CONCLUSION

From thermomechanical analysis is clear that due to various thermal histories the results for basalt rods and composites from filaments roving are not directly comparable. Glass transition temperature of fibers 526.6°C is about 70°C lower that the T_g of rods. The linear thermal expansion coefficients are comparable with published results (about $7.7\,10^{-6}$ deg^{-1}). The maximum dilatation D grows with temperature slowly and the half time of dilatation exhibits no trend. Longitudinal compressive creep modulus of Basalt is relatively high and temperature insensitive in investigated region. The main problems is definition of initial deformation. We have used the deformation $\varepsilon_{(10)}$ after 10 sec. of compressive creep to prevent initial uncertainty of measurement.

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