THERMOPHYSICAL AND MECHANICAL PROPERTIES OF FIBER-REINFORCED COMPOSITE MATERIAL SUBJECTED TO HIGH TEMPERATURES

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Abstract. The bulk density, open porosity, matrix density, tensile strength, bending strength, thermal diffusivity, specific heat capacity, thermal conductivity and linear thermal expansion coefficient of high-density glass fiber reinforced cement composite are determined as functions of temperature up to 1000 °C. The basic physical parameters and mechanical parameters are found to exhibit the most important changes between the reference state and 600 °C pre-heating where the increase of porosity is as high as 40% and both the tensile strength and bending strength decrease to about one third of their original values. The measured dependences of thermal diffusivity and thermal conductivity on temperature indicate that the heat transfer in the studied material is accelerated once temperature achieves 500–600 °C but the change in heat storage expressed by the specific heat capacity is less important. The linear thermal expansion coefficient is not found to be affected by high temperatures in a negative way; it is either lower or comparable to its low-temperature values.

Keywords: glass-fiber reinforced cement composites, high temperatures, tensile strength, bending strength, thermal diffusivity, specific heat capacity, thermal conductivity, linear thermal expansion coefficient.

1. Introduction

Glass-fiber reinforced cement composites (GFC) were developed to improve the tensile and flexural strength of inorganic cements. Since the beginning of 1970s they have found their place as versatile and commercially viable materials for use in construction industry with a wide application range from façade panels to formwork, pipework, and even surface bonding and rendering (Bentur and Mindess 1990; Majumdar and Laws 1991). Therefore, their mechanical performance always was of primary concern of both researchers and building industry. This was also reflected in a variety of scientific papers dealing with measurements of mechanical properties during the last at least 40 years. These were most numerous in 1970s and 1980s when the development of production technology of GFC was the fastest. Within the last ten years, the number of references describing measurements of mechanical properties of GFC decreased, as compared to 1970s and 1980s. Nevertheless, still many interesting new experimental results were presented by various scientists all over the world. Zhang *et al.* (1997) measured flexural strength of GFC with high content of fly ash, Marikunte *et al.* (1997) determined the flexural and tensile strength of GFC after hot-water durability tests, Park *et al.* (1999) measured compressive strength, compressive modulus of elasticity, tensile strength, flex-

ural strength of low-density/low cost GRC, Trtik and Bartos (1999) studied the fracture mechanisms in GFC using in-situ SEM bending tests, Purnell *et al.* (1999) carried out mechanical tests of GFC after various accelerated aging procedures, Mu *et al.* (2000) determined tensile strength and impact resistance of short fiber GRC with high slag content, Purnell *et al.* (2000) measured the bond strength in GFC using petrography and image analysis, Purnell *et al.* (2001a) studied the effects of super-critical carbonation on mechanical properties, Seneviratne *et al.* (2002) examined the possibilities to reduce the swelling and shrinkage of GFC under various environments using super-critical carbonation, Purnell and Beddows (2005) studied the strength loss of new formulations of GFC in accelerated aging using the static fatigue model formulated by Purnell *et al.* (2001b), Cuypers *et al.* (2006) analyzed the durability of GFC on the basis of monotonic and cyclic tensile loading, Barluenga and Hernandez-Olivares (2007) used short glass fibers in concrete at early age to improve cracking control.

The mechanical properties will certainly remain the principal parameters for GFC design and technology also in the future. However, sometimes a one-sided approach to the performance assessment of GFC elements in building structure may not be satisfactory. There are numerous GFC applications where determination of just mechanical

properties is not sufficient and thermal and hygric properties may become significant, or even more important. To give only couple of examples, façade panels are supposed to meet certain thermal and hygric requirements, lightweight GFC can be used as thermal insulation materials, some other types of GFC can be used as fire protection materials to replace previously used asbestos-cement products (West *et al.* 1980a, b). In the first two cases thermal and hygric properties should be known in sufficient temperature and moisture ranges to determine the hygrothermal performance of the wall systems, in the third the high-temperature thermal properties such as thermal conductivity, specific heat capacity and linear thermal expansion coefficient are supposed to be measured to assess the fire-protection function of the envelope in an appropriate way. Despite this apparent need for a more general consideration of the properties of GFC in material and structural design, the references giving results of determination of thermal and hygric properties of GFC are still quite rare, as compared to the measurements of mechanical properties. The papers by Černý *et al.* (2004) and de Guttierez *et al.* (2005) belong to the very few exceptions in that respect.

In this paper, a complex view on the properties of GFC is presented which involves a combination of thermophysical and mechanical parameters that was not considered in previous references, not even in the relatively extensive paper by Černý *et al.* (2004) where three GFC of different composition were studied. Besides the measurement of tensile and bending strength also the principal thermal properties, namely the thermal diffusivity, specific heat capacity, thermal conductivity and linear thermal expansion coefficient, of high-density glass fiber reinforced cement composite are determined as functions of temperature up to 1000 °C. The relations between the changes in mechanical and thermal parameters due to the high-temperature exposure are discussed as well.

2. Experimental methods

2.1. Basic physical properties

The bulk density, matrix density and open porosity were measured using the water vacuum saturation method (Roels *et al.* 2004). Each sample was dried at first in a drier to remove majority of the physically bound water and the mass of dry sample was determined. After that the samples were placed into a desiccator with deaired water. During three hours air was evacuated with vacuum pump from the desiccator. The samples were then kept under water not less than 24 hours. Finally, the mass of water saturated sample and immersed water saturated sample were measured, the volume of the sample was determined, and the open porosity, bulk density and matrix density were calculated.

2.2. Tensile strength and bending strength

The measurement of tensile strength was done using the electromechanical testing device MTS Alliance RT 30 with the maximum tension force of 30 kN. The rate of loading was 0.025 mm/min. The bending strength was measured in the same device provided with the three-point bending track 642.01A which allowed the maximum load of 27.80 kN. The rate of loading was 0.04 mm/min. The values of tensile strength and bending strength were calculated from the measured force values using the common physical formulas.

2.3. Thermal diffusivity

For the determination of high-temperature thermal diffusivity the double integration method by Drchalová and Černý (1998) was used which was developed originally for moisture transport. For heat conduction equation it can be applied with minor changes only. The final equation for the calculation of thermal diffusivity a reads

$$a(\tau) = \frac{\displaystyle\int_0^{x_0(\tau,t_n)} I_T(T(x,t_n))\,dx - \int_0^{x_0(\tau,t_1)} I_T(T(x,t_1))\,dx}{\displaystyle\int_{t_1}^{t_n} \frac{\partial T}{\partial x}(x_0(\tau,t),t)\,dt} -$$

$$\frac{I_T(\tau)[x_0(\tau,t_n) - x_0(\tau,t_1)] - \displaystyle\int_{t_1}^{t_n} \frac{j_Q(0,t)}{\rho(\tau)c(\tau)}\,dt}{\displaystyle\int_{t_1}^{t_n} \frac{\partial T}{\partial x}(x_0(\tau,t),t)\,dt}, \qquad (1)$$

where $T(x,t)$ is the temperature field in the sample measured at one-sided heating, τ is a chosen constant value of temperature, $x = x_o(\tau,t)$, $t = t_o(\tau,x)$ are one-to-one parametrizations corresponding to τ ($T(t)$ and $T(x)$ are supposed to be monotonic functions, both x_o and t_o are monotonic functions as well), t_1, t_n are the times corresponding to the first and last measured temperature profile, respectively, c is the specific heat capacity, ρ is the bulk density:

$$I_T(T) = \int \frac{\partial T}{\partial t}\,dt = \int dT, \qquad (2)$$

and the heat flux at $x=0$, $j_Q(0,t)$, can be for $t_j > t_i$ calculated as

$$j_Q\left(0, \frac{t_j + t_i}{2}\right) =$$

$$\frac{1}{t_j - t_i} \int_0^D \left[\rho(T)c(T)T(x,t_j) - \rho(T)c(T)T(x,t_i) \right] dx, \qquad (3)$$

where D is the length of the one-dimensional domain under consideration.

In the measuring procedure itself, one-side heating of a specimen with thermally insulated lateral faces is realized using a furnace where a constant temperature is maintained. Along the longitudinal axis of the sample, a set of temperature sensors is positioned, which makes it possible to record the temperature field through a measuring unit by a PC. From the measured $T(x,t_i)$ curves, a set

of 8–10 curves is chosen, and these curves are used in the computational treatment. First, the measured $T(x,t_i)$ curves are subject of a regression analysis. Then, we choose a temperature value τ, determine the integration area for this value and calculate the corresponding value of thermal diffusivity $a(\tau)$ from Eq. (1). This procedure is repeated for a sufficient number of τ values so that we finally obtain a point-wise given function $[\tau_i, \ a(\tau_i)]$.

2.4. Specific heat capacity

As the adiabatic methods are not very suitable for measuring high-temperature specific heat capacity of building materials, mainly because of the necessity to use relatively large samples, a nonadiabatic method (Toman and Černý 1995) was employed for the determination of temperature-dependent specific heat capacity. The nonadiabatic calorimeter designed in Toman and Černý (1995) has a mixing vessel with a volume of 2.5 liters. The volume of the measuring fluid (water in this case) is about 1 liter. The maximum volume of the measured samples is 1 liter. The amount of heat loss of the nonadiabatic system is determined using a calibration. The calorimeter is filled with water, whose temperature is different from the ambient air. Then, the relation of water temperature to time, i.e., the calibration curve is measured.

The measuring method itself is based on well-known principles. The sample is heated to a predetermined temperature T_s in a furnace and then put into the calorimeter with water. Then, the relation of water temperature to time, $T_w(t)$, is measured, water being slowly stirred all the time, until the temperatures of the measured sample and the calorimeter are equal. The corrected (adiabatic) temperature T_e taking the heat loss into account is calculated and substituted into the heat balance equation of the sample-calorimeter system:

$$mc(T_s - T_e) = (K + m_w c_w)(T_e - T_{wo}) + \Delta m_{evap} \cdot L_w - Q_r, \tag{4}$$

where m is the mass of the sample, c is the specific heat capacity of the sample in the temperature interval $[T_e, T_s]$, K is the heat capacity of the calorimeter, m_w is the mass of the water, c_w is the specific heat capacity of water, T_{wo} is the initial water temperature, L is the latent heat of evaporation of water, Q_r is the reaction heat, Δm is the mass of evaporated water.

Dividing both sides of eq. (4) by m and specifying the zero-point of the enthalpy scale (see Toman and Černý 1995, for details), we can calculate the value of specific enthalpy corresponding to the furnace temperature T_s, $h(T_s)$. The specific heat capacity can then be determined using its definition relation:

$$c(T_i) = \frac{\partial h}{\partial T}(T_i). \tag{5}$$

Performing a set of measurements for various sample temperatures T_i, we obtain a set of points $[T_i, h(T_i)]$. A regression analysis of this point-wise given function results in a functional relationship for $h = h(T)$ and, using

eq. (5), also in the $c = c(T)$ function as the first derivative of h with respect to T.

2.5. Linear thermal expansion coefficient

The high-temperature measurements of linear thermal expansion coefficient α were performed using the method proposed in Toman *et al.* (1999). The measuring device is based on the application of a comparative technique. In the practical determination of the linear thermal expansion coefficient the measured sample and the standard are put into the furnace, provided with contact ceramic rods, and the initial reading on the digital dial indicators is taken. Then, the electric heating regulation system is adjusted for the desired temperature T_i in the furnace, and the length changes are monitored. The data acquisition from the digital dial indicators is done on PC using specially developed software. After the steady state is achieved, i.e., no temperature changes in the furnace and no length changes of both measured sample and the standard are observed, the final readings of length changes are taken. The length change of the measured sample itself is then calculated and the corresponding value of thermal strain, $\varepsilon(T_i)$, is determined. The measurements are repeated with other chosen values of furnace temperatures T_i, the regression analysis of the point-wise given $[T_i, \varepsilon(T_i)]$ function results in a functional relationship for $\varepsilon = \varepsilon(T)$, and finally the calculation of the $\alpha(T)$ function of the measured material is performed using the definition relation

$$\alpha = \frac{d\varepsilon}{dT}. \tag{6}$$

3. Materials and samples

The measurements were done on a glass fiber reinforced cement composite produced in the laboratories of VUSTAH Brno. The composition of the material was as follows (calculated among the dry substances only): Portland cement CEM I 52.5 54%, sand 0–1 mm 40%, microsilica 3%, alkali-proof glass fiber 12 mm long 3%. Water in the amount corresponding to the w/c ratio of 0.33 was added to the mixture.

The samples were produced using a successive homogenization procedure. At first, sand and microsilica were homogenized in a mixing device, then cement was added and the dry mixture was homogenized again. The dry well homogenized mixture was thoroughly mixed with water. Finally, the glass fibers were added and the mixture shortly mixed again. The prepared mixture was cast into the molds and vibrated. After the time period of 28 days after mixing, the samples were prepared for testing.

In the experimental measurements, four various sample pre-treatment conditions were tested: reference specimen not exposed to any load (denoted as UM in what follows), specimen exposed to a gradual temperature increase up to 600, 800 and 1000 °C during two hours, then left for another 2 hours at the final tempera-

ture and slowly cooled (denoted as UM-600, UM-800 and UM-1000 according to the loading temperature).

The samples for the measurement of particular material parameters had the following dimensions: basic physical properties – $30\times30\times10$ mm, tensile strength – $20\times10\times160$ mm, bending strength – $40\times40\times160$ mm, thermal diffusivity and specific heat capacity – $71\times71\times71$ mm, linear thermal expansion coefficient – $40\times40\times120$ mm. For every pre-treatment, 3 specimens were used for testing. Before the measurements of all parameters, all specimens were dried in an oven at 110 °C.

4. Experimental results and discussion

Table 1 shows basic physical parameters of the studied material depending on the loading temperature. The most important change in porosity occurred between the unloaded state and the loading temperature of 600 °C where the increase of porosity was as high as 40%. Later porosity changes were lower than 10%. This is in accordance with the behavior of most Portland cement based composite materials where the most important chemical reaction in the high temperature range is decomposition of calcium hydroxide at about 460–480 °C (see Černý *et al.* 2003, for more details).

**Table 1.** Basic physical parameters

Material	Open porosity $[m^3\,m^{-3}]$	Bulk density $[kg\,m^{-3}]$	Matrix density $[kg\,m^{-3}]$
UM	0.25	1872	2491
UM-600	0.35	1885	2898
UM-800	0.38	1822	2929
UM-1000	0.38	1812	2903

Table 2 presents tensile strength and bending strength of the particular groups of specimens. Similarly as in the case of basic physical parameters, the most important changes in mechanical properties were observed between the reference state and the specimens loaded to 600 °C, where both tensile strength and bending strength decreased to about one third of their original values. UM-1000 specimens were not investigated because they disintegrated during insertion into the testing device, so that their mechanical properties could not be determined.

Table 2. Tensile strength and bending strength

Material	Tensile strength [MPa]	Bending strength [MPa]
UM	2.37	15.00
UM-600	1.00	4.62
UM-800	0.22	2.57
UM-1000	–	–

Fig. 1 shows the results of thermal diffusivity vs. temperature measurements. In the lower temperature range, the values of thermal diffusivity increased relatively moderately with increasing actual temperature but this increase became quite steep after achieving 600 °C. This feature was observed for all pre-heating temperatures. The most probable reason for these findings was an
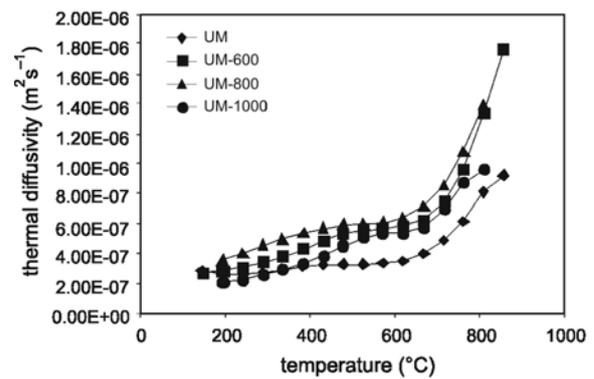
Fig. 1. Thermal diffusivity as a function of temperature

inclusion of the convective and radiative modes of heat transfer into the values of thermal diffusivity which, due to the transient character of measurements and the occurrence of high temperature gradients in the measured specimens, was not a common thermal diffusivity in Fourier's sense but an apparent thermal diffusivity. From a practical point of view this seems to be a positive factor because any appearance of high temperatures in a building structure is often accompanied by high temperature gradients, which are not always accounted for properly in the fire-safety considerations. Using the values of apparent thermal diffusivity in the commonly utilized simple computer codes working with Fourier heat conduction can include the convective and radiative modes of heat transfer into the fire-safety related calculations at least in an empirical way, thus increase the physical correctness of the numerical analysis. The thermal diffusivity of specimens pre-heated to 600 °C was up to two times higher, as compared to the reference specimens, but for higher pre-heating temperatures any further significant increase was not observed. The differences were within the error range of the applied measuring method. This is in a good accordance with the measurements of open porosity in Table 1.

Fig. 2 shows the measured dependence of specific heat capacity on temperature. In the low-temperature range, up to approximately 300 °C, an increase of specific heat capacity was observed but for higher temperatures the changes were very low; the specific heat capacity remained almost constant. The small decrease of specific heat capacity for temperatures higher than 400 °C can be attributed to the partial loss of mass due to the decomposition of calcium hydroxide.

Fig. 3 presents the thermal conductivity as function of temperature which was calculated on the basis of measured thermal diffusivity, specific heat capacity and bulk density. Clearly, the shapes of the $\lambda(T)$ functions were very similar to those of $a(T)$ functions. This is a consequence of the more significant dependence of thermal diffusivity on temperature in a comparison with the specific heat capacity and bulk density.

The results of measurements of the linear thermal expansion coefficient in Fig. 4 showed that in the range of lower temperatures the measured data corresponded well with the common α values known for cement mortar and concrete (Toman *et al.* 1999; Toman and Černý

2001) and also for higher temperatures the observed changes were not very dramatic. The minima at approximately 500 °C and 800 °C are most probably a consequence of decomposition reactions in the cement binder, namely calcium hydroxide in the first case and calcium carbonate in the second (Toman *et al.* 1999).
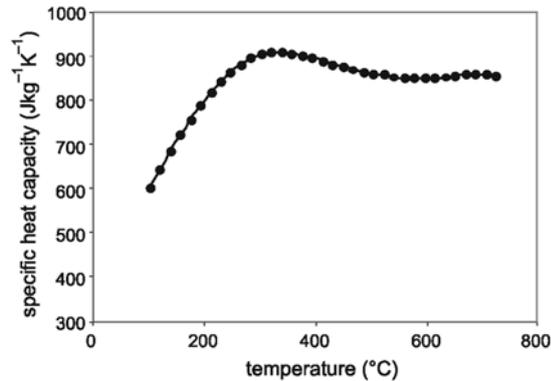

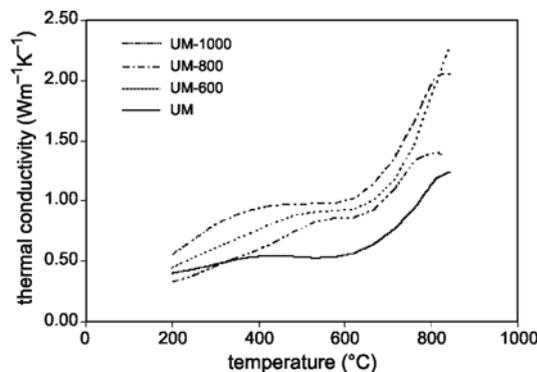**Fig. 2.** Specific heat capacity as a function of temperature

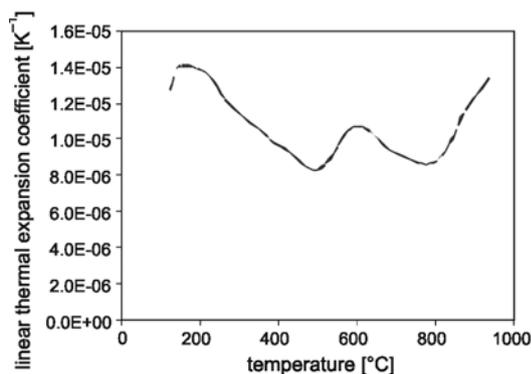**Fig. 3.** Thermal conductivity as a function of temperature

Fig. 4. Linear thermal expansion coefficient as a function of temperature

5. Conclusions

The measurements of temperature dependence of mechanical and thermal properties of the glass-fiber reinforced cement composite in this paper have shown that the decomposition of calcium hydroxide was the principal factor affecting the behavior of thermally loaded specimens. The basic physical parameters and mechanical parameters exhibited the most important changes between

the reference state and the 600 °C pre-heating where the increase in porosity was as high as 40%, and both the tensile strength and bending strength decreased to about one third of their original values. The effect of decomposition of calcium carbonate was significant for the linear thermal expansion coefficient only.

For the fire protection of building structures, the measured dependences of thermal diffusivity and thermal conductivity on temperature indicated that the heat transfer in the studied material was accelerated once temperature achieved 500–600 °C which might be dangerous because the fire-protecting ability of such a structure decreases in a comparison to the values calculated with data measured at lower temperatures. Using the high-temperature data in the evaluation of fire-protecting abilities of such structures is therefore highly recommended. On the other hand, the thermal expansion of the analyzed GFC did not increase in the high-temperature range in a significant way, and therefore the real thermal stress would not be much higher compared to the calculations with room temperature data which is a positive information.

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TERMOFIZINĖS IR MECHANINĖS PLUOŠTU SUSTIPRINTO KOMPOZITO, KURĮ VEIKIA AUKŠTA TEMPERATŪRA, SAVYBĖS

E. Vejmelková, P. Konvalinka, P. Padevět, R. Černý

S a n t r a u k a

Tiriama, kaip kinta tankiojo stiklo pluoštu armuoto cementinio kompozito tankis, atvirasis poringumas, matricinis tankis, tempiamasis bei lenkiamasis stipris, terminis laidumas, savitasis šilumos imlumas, savitasis šilumos laidumas ir tiesinis terminio plėtimosi koeficientas, kai šią medžiagą veikia temperatūra, kylanti iki 1000 °C. Rasta, kad pagrindiniai fiziniai ir mechaniniai parametrai daugiausia keičiasi temperatūrai kylant nuo pradinės iki 600 °C. Šioje temperatūros kilimo atkarpoje poringumas išaugo iki 40 %, o tempiamasis ir lenkiamasis stipriai sumažėjo maždaug trečdaliu, palyginti su pradinėmis reikšmėmis. Išmatuotoji terminio laidumo ir savitojo šilumos laidumo priklausomybė nuo temperatūros rodo, kad šilumos perdavimas tiriamoje medžiagoje pagreitėja temperatūrai pasiekus 500–600 °C, tačiau šilumos kaupimas, išreikštas savituoju šilumos imlumu, yra ne toks svarbus. Nenustatyta, kad tiesinis terminio plėtimosi koeficientas būtų neigiamai veikiamas aukštos temperatūros. Šio koeficiento reikšmės yra mažesnės arba maždaug lygios reikšmėms, išmatuotoms žemoje temperatūroje.

Reikšminiai žodžiai: stiklo pluoštu stiprinti cemento kompozitai, aukšta temperatūra, tempiamasis stipris, lenkiamasis stipris, terminis laidumas, savitasis šilumos imlumas, savitasis šilumos laidumas, tiesinis terminio plėtimosi koeficientas.

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