

Primljen / Received: 26.11.2013.

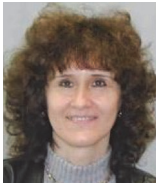
Ispravljen / Corrected: 4.6.2014.

Prihvaćen / Accepted: 14.8.2014.

Dostupno online / Available online: 10.11.2014.

Measurement of physical properties of polyurethane plaster

Authors:



Assoc.Prof. **Milena Kušnerová**, PhD. Physics
VŠB - Technical University of Ostrava
Faculty of Mining and Geology
Faculty of Metallurgy and Materials
milena.kusnerova@vsb.cz



Assoc.Prof. **Jan Valíček**, PhD. Physics
VŠB - Technical University of Ostrava
Faculty of Mining and Geology
Faculty of Metallurgy and Materials
jan.valicek@vsb.cz



Marta Harničárová, PhD. ME
VŠB - Technical University of Ostrava
Faculty of Mining and Geology
Faculty of Metallurgy and Materials
marta.harnicarova@vsb.cz

Preliminary note

Milena Kušnerová, Jan Valíček, Marta Harničárová

Measurement of physical properties of polyurethane plaster

Physical properties of building materials can be defined by material coefficients. The material coefficients examined were: coefficient of mean density, coefficient of specific heat capacity, coefficient of thermal conductivity, and coefficient of thermal diffusivity. Physical properties of a newly-developed material (polyurethane plaster), were subjected to non-destructive analysis. The same method and same conditions of measurement were applied to measure the properties of comparative samples made of Ytong. In order to measure the coefficient of thermal conductivity and coefficient of thermal capacity, a new comparative method was designed and verified and also a new calorimetric apparatus was used.

Key words:

polyurethane plaster, new method of measurement, specific heat capacity, thermal conductivity, thermal diffusivity

Prethodno priopćenje

Milena Kušnerová, Jan Valíček, Marta Harničárová

Mjerenje fizikalnih svojstava poliuretanske žbuke

Fizikalna svojstva građevnih materijala mogu se definirati pomoću koeficijenta materijala kao što su srednja gustoća, specifični toplinski kapacitet, toplinska provodljivost i toplinska difuzivnost. U radu je prikazano ispitivanje fizikalnih svojstava novorazvijenog materijala (poliuretanske žbuke) nerazornim postupkom. Također je provedeno određivanje svojstava komparativnih uzoraka izrađenih od porobetona pri čemu je primijenjena ista metoda kao i isti uvjeti ispitivanja. Za određivanje toplinske provodljivosti i specifičnog toplinskog kapaciteta razvijena je i verificirana nova komparativna metoda za čije je provođenje razvijeni novi kalorimetrijski uređaj.

Ključne riječi:

poliuretanska žbuka, nova metoda mjerenja, specifični toplinski kapacitet, toplinska vodljivost, toplinska difuzivnost

Vorherige Mitteilung

Milena Kušnerová, Jan Valíček, Marta Harničárová

Messung physikalischer Eigenschaften von Polyurethanpflaster

Physikalische Eigenschaften von Baumaterialien können mittels Materialkoeffizienten, beispielsweise der durchschnittlichen Dichte, der spezifischen Wärmekapazität, der Wärmeleitfähigkeit und der Wärmediffusivität, definiert werden. In dieser Arbeit sind Untersuchungen der physikalischen Eigenschaften eines neu entwickelten Materials (Polyurethanpflaster) durch zerstörungsfreie Messungen dargestellt. Des Weiteren sind die Eigenschaften an Vergleichsproben aus Porenbeton unter gleichbleibenden Versuchs- und Messbedingungen ermittelt worden. Um die Wärmeleitfähigkeit und die spezifische Wärmekapazität zu messen, ist ein neues Vergleichsverfahren eingeführt und verifiziert worden, für das ein neues kalorimetrisches Messgerät entwickelt wurde.

Schlüsselwörter:

Polyurethan-Putz, spezifische Wärmekapazität, Wärmeleitfähigkeit, Wärmediffusivität, neue Messmethode

1. Introduction

Polyurethane plaster is a new type of heat-insulating plaster designed for use on the external and internal walls of buildings. Polyurethane foam in form of crumbs with the maximum grain diameter of 4 mm is used, at the end of its life cycle, as an extender. The proper treatment of the hard polyurethane foam after completion of its life cycle, and an example of application of heat-insulating polyurethane-based plaster to a reference structure, is presented in [1-3]. The thermal conductivity of solids is studied as an interchange of heat by conduction, either as a steady/stationary heat conduction, or as a time variable/non-stationary heat conduction. The heat exchange is generally defined as an imbalanced thermodynamic process, which takes place under the condition involving temperature difference. The thermal flow of a solid is then defined as the thermal flow that travels from the locality of a studied material with higher temperature, to the locality with lower temperature. The heating process starts with the phase of non-stationary conduction, and continues with the stationary phase when the material reaches a certain heat capacity. The steady thermal flow is defined as the thermal flow that takes place at a constant temperature difference. The relationships between physical parameters of the thermal conduction process are expressed by Fourier's law. According to Fourier's law, the density of thermal flow is directly proportional to thermal gradient. The material coefficient derived from this direct proportion is called the specific thermal conductivity coefficient. The specific heat capacity is studied using calorimetric methods. The sample of the studied material is supplied with heat by electric heating in a calorimeter, which is in principle a heat-insulated vessel. The relationships between physical parameters of the heat capacity process are expressed by the calorimetric equation.

The current state of the problems studied is related to the traditional methods of measurement, and the study of thermal properties of materials. The thermal conductivity is studied as an interchange of heat by conduction in a given material. The material coefficient of thermal conductivity, both at the steady (stationary) and time-variable (non-stationary) heat conduction, has an informative value with regard to material properties. According to Fourier's law, the heat conducted through the studied material is measured between parallel specimens; this heat is directly proportional to the temperature difference between both specimens, area of the specimens, and the time of heating, and is indirectly proportional to the distance between them. Stationary contact methods for the measurement of heat conduction are more demanding as to apparatuses, and the time of measurement is relatively longer when compared to the contact non-stationary methods of measurement. However, these measurements are highly accurate and are therefore often regarded as standard measurements (under exactly declared conditions because the reliability of measurement results is associated with the width of temperature interval; the sensitivity and resolution of apparatuses are connected with the potential change of material homogeneity and moisture).

In addition, the heat is studied as a part of change of the inner energy that is interchanged by the material in contact with another material of different temperature. In this case, it is the coefficient of specific heat capacity that has an informative value with regard to material properties. The specific heat capacity is measured according to the traditional calorimetric method and, at that, the heat needed to change the sample temperature – which is directly proportional to the mass of material – is measured.

Apparatuses that are designed for the measurement of thermal coefficients (i.e. for the measurement of thermal conductivity and specific heat capacity) enable direct measurements, which especially concerns the density of thermal flow and temperature difference measurements.

Traditional but still widely used apparatuses are based on stationary contact methods. They namely include all plate-based apparatuses according to Poensgen and Bock, cylinder apparatuses according to Van Rinsum, and spherical apparatuses according to Nusselt.

Modern apparatuses in common use are based on non-stationary contact methods, and are simple, fast, relatively accurate, and computer-controlled. In principle, these are apparatuses according to Fitch, where the difference in temperature between two material localities is measured in the regular phase over a certain period of time. For instance, a very well-known apparatus Thermophysical Transient Tester RT is based on a pulse method (a heating pulse is emitted to the measured material, and a sensor recording the thermal response for a certain time is placed behind the material). The apparatus Shotherm QTM makes use of the hot wire method (a temperature rise is measured within the defined distance from the linear source of heat placed inside the measured material).

Latest methods for the measurement of thermal coefficients are the contactless methods in which infrared sensors are used. This particularly concerns the CFTT-05 apparatus that uses contactless flash methods (measured current temperature values are sensed by an infrared sensor placed behind the irradiated material). Although these new methods of measurement are fast, their results are less accurate in comparison with traditional contact methods.

The following traditional physical relations should be used as a basis for derivation of new theoretical relations (1) to (4) for calculating thermal characteristics of a new insulating material (polyurethane) using the newly proposed method and equipment.

a) Mean density ρ (volume density in dry state):

$$\rho = \frac{m}{S \cdot d} \quad (1)$$

where is m - the mass of the board, S - the area of the board wall, and d - the board wall thickness [4].

b) According to the law of conservation of energy, the coefficient of specific heat capacity c amounts to:

$$Q = m \cdot c \cdot \Delta t \quad (2)$$

where Q is the heat supplied to the lower base of the board by heating, m is the mass of the board, Δt is the change of temperature of a materia [4].

- c) The coefficient of thermal conductivity λ is defined according to Fourier's law as follows (3)

$$Q = \lambda \cdot \frac{S}{d} \cdot \Delta t \cdot \tau \quad (3)$$

where Q is the heat supplied, mostly by contact heating, to a regularly shaped sample (e.g. a board), while Δt is the measured time of heat flow through the material of the board [4, 5]. S is the sample wall area, and d is the sample thickness. The difference in temperature between the external heating and cooling is constant. Two probes are nested into the grooves in the upper and lower wall of the board at a distance d so that the thermometer measures the temperature difference Δt between the two locations inside the sample material. However, this temperature difference in the sample material is not constant throughout the heating process. The heating in the studied material takes place in three stages.

In the first stage of the heating process, at the beginning of the measurement, the value of the temperature difference Δt is zero, but it gradually increases depending on the intensity of the heat, since the board is heated at a varying non-stationary heat flow. The period of the stationary heat flow Δt cannot as yet be measured.

In the second stage of the heating process, the temperature difference Δt becomes a certain constant value, since the board conducts heat without further heat absorption. The heat flow through the material has already become stationary, and so the temperature difference Δt is suitable for accurate calculation of the coefficient λ . The period of stationary heat flow Δt is limited by the remaining five physical quantities ($Q, \lambda, S, d, \Delta t$).

The temperature difference Δt begins to decrease in the third stage of the heat action, and the board overheats. The heat flow through the material becomes non-stationary again. The measured temperature difference varies and oscillates, and so measurement results are inappropriate for accurate calculation of the coefficient λ , just like in the first stage of the heating process. The period of stationary heat flow Δt is completed.

The disadvantage of this method is that the heating heat must be accurately determined (with regard to the heat loss to the outside environment), and that the constant heating and cooling difference must be maintained. This is a traditional laboratory method; the apparatus is relatively expensive and challenging to any mobility. However, it results in a very good prediction of measurement results, which is an advantage.

It should also be noted that the commonly used tables show the coefficient λ value at room temperature (20 °C). This material coefficient is also slightly dependent on the temperature and moisture of material. If there is a need for very accurate coefficient λ results for an unknown material, it is always necessary to specify the mean temperature t_s , at which the stationary heat flow passes through the material ($t_s = t_1 + \Delta t/2$).

The newly proposed method is obviously also based on the Fourier's law of heat conduction, and therefore, the heat action

must take place in three above-mentioned stages: non-stationary, stationary and once again non-stationary heat flow.

With regard to the complexity and accuracy that are needed to determine the coefficient λ , the advantage of the newly proposed measurement method lies in the fact that the amount of the heating heat need not be controlled, which also applies to the heat loss to external environment. Furthermore, it is not necessary to maintain a constant heating and cooling temperature difference at opposite sides of the sample. Concerning the complexity and calculation accuracy needed to determine the coefficient λ , the disadvantage of the newly proposed measurement method lies in the need to obtain very accurate information about the coefficient λ for the reference board, and about its temperature development. The next step is the comparison between the measurement results for heat action taking place in the known reference sample, and heat action taking place in an unknown studied sample (comparison results can be saved to a computing program). It should be noted that the measurements must be performed on the same device and under the same conditions. According to equation (6), only two periods of stationary heat flow, and both thermal conductivity coefficient values, will need to be compared in this case.

In order to adequately slow down the heating action (i.e. to enable accurate measurement), the measured board (known reference and samples) is inserted into the system of other boards forming a "sandwich", where boards are arranged in a particular and exactly specified order which is unchangeable.

In the first stage of the heating action, at the beginning of measurement, the value of temperature difference is zero but, depending on the intensity of heat, it gradually increases, and the board is heated at a varying non-stationary rate of heat flow. In the second stage of the heating action, the temperature difference Δt acquires its maximal and relatively constant value for the period Δt . Then the stationary heat flow passes through the board material. Therefore, the constant value of the temperature difference Δt between the sensed temperatures in the grooves of the opposite boards can be used for accurate calculation of the coefficient λ . In the third stage of the heating action, the temperature difference Δt begins to decrease. Unless the heat action is finished, the system of the boards starts to overheat as a whole. The heat flow through the material is non-stationary; the difference in temperature is variable, and oscillating. The results of measuring the temperature difference Δt are inaccurate and inappropriate for proper calculation of the coefficient λ analogously as in the first stage of the heating action.

- d) The coefficient of thermal diffusivity a is defined as a correlation between Eqs. (1), (2), (3) and, with a physical meaning of speed of temperature change during thermal conduction in a quasi-homogenous environment, it is defined according to [4, 5] as follows:

$$a = \frac{\lambda}{\rho \cdot c} \quad (4)$$

When the conditions of both measurements are the same, we can assume that the values of heat supplied to the comparative board Q_1 and the studied board Eq. Q_2 (2) are approximately the same:

$$Q_1 = Q_2 \Rightarrow c_2 = c_1 \cdot \frac{\Delta t_1 \cdot m_1}{\Delta t_2 \cdot m_2} \tag{5}$$

The unknown coefficient of specific heat capacity c_2 Eq. (5) is measured using m_1 , which is the mass of the comparative board, Δt_1 is the temperature difference between the walls of the comparative board, Δt_2 is the temperature difference between the walls of the studied board, and c_1 is the known value of the specific heat capacity coefficient of the comparative board.

When the conditions of both measurements are kept the same, we can assume that the heat supplied to the comparative sample Q_1 and the studied sample Q_2 Eq. (3) are approximately the same, which means that the input power P of the calorimetric apparatus is the same, as well as the total time of measurement τ . The area S of the boards, the efficiency η of the calorimetric apparatus, and the heat K absorbed by other components of the calorimetric apparatus, are also the same.

$$P \cdot \tau \cdot \eta = \lambda_1 \cdot \frac{S}{d_1} \cdot \Delta t_1 \cdot \tau + K \wedge P \cdot \tau \cdot \eta = \lambda_2 \cdot \frac{S}{d_2} \cdot \Delta t_2 \cdot \tau + K \Rightarrow \lambda_2 = \lambda_1 \cdot \frac{d_2}{d_1} \cdot \frac{\Delta t_1}{\Delta t_2} \tag{6}$$

The unknown coefficient of thermal conductivity λ_2 Eq. (6) is measured using the temperature difference, Δt_1 between walls of the board of the comparative material, Δt_2 temperature difference between the walls of the board of the studied new material, and λ_1 which is the known coefficient of thermal conductivity of the comparative board. The values of d_1 , which is the thickness of the comparative board, and d_2 which is the thickness of the studied board, are the same.

2. Experimental part

30 test specimens of the studied polyurethane plaster and 10 comparative specimens of ytong (P2-500, reference material) were modified into boards of equal geometrical proportions (10 x 10 cm). The board thicknesses were 1 cm, 2 cm, 3 cm, 4 cm, 5 cm, 6 cm, 8 cm, and 10 cm. The thicknesses of each pair of samples (studied board and comparative board) were always the same. Non-destructive methods of measurement were chosen so as not to change the insulating and physical properties of the material. The samples were then weighed, and their geometrical proportions were measured. These processes took place simultaneously with the measurement of temperature difference between their walls. The material of the studied samples was quasi-homogenous and porous, and so a highly accurate approach was needed in the measurement of material coefficients. Each sample of either studied or comparative board was measured under strictly defined and comparable conditions. The temperature difference between sample walls was measured under steady heating in equidistant time intervals, either during steady or unsteady thermal flow. The measurements were conducted using devices complying with requirements applied in engineering practice (temperature determination accuracy 2.5 %, and time determination accuracy 0.2 s). Measurement results were evaluated based on the valid methodology for assessing mean values of measurement results and their uncertainties. The

accuracy of results was defined using both absolute and relative uncertainties. The proposed apparatus (Figures 1, 2) was used to perform basic measurements, while subsidiary measurements were conducted using devices commonly available in engineering practice (analytical balance and electronic beam calliper) [6]. Test specimen is shown in Figure 3.

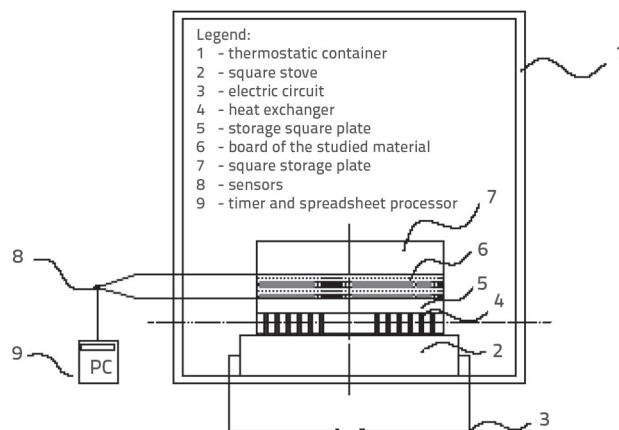


Figure 1. Scheme of calorimetric apparatus



Figure 2. Calorimetric apparatus prototype

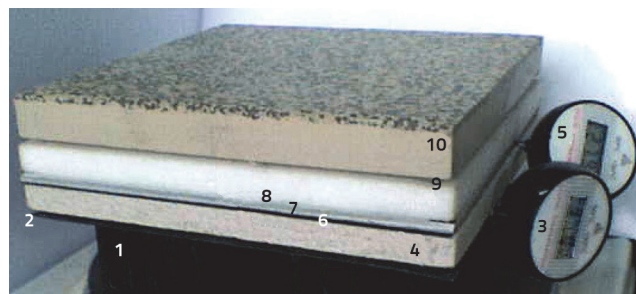


Figure 3. Test specimen in the apparatus: 1-metal plate of heat source, 2-thin plastic board, 3-control probe of thermometer, 4-measured polyurethane board, 5-control probe of thermometer, 6-thin plastic board, 7-thick plastic board, 8-thin metal board, 9-thick polystyrene board, 10-thick stoneware load board

Table 1. Part of measurement values for defining the thermal conductivity coefficient for polyurethane

Number of measurement	Δt_1 [°C]	Δt_2 [°C]	λ_1 [W/mK]	λ_2 [W/mK]	$\Delta \lambda_2$ [W/mK]	$(\Delta \lambda_2)^2$ [W ² /m ² K ²]	u_{λ} [W/mK]
18	71,8	71,5	0,12	0,120503	-0,00061283	$3,75556 \cdot 10^{-7}$	
19	71,9	72	0,12	0,119833	5,73366E-05	$3,28749 \cdot 10^{-9}$	
20	71,8	72,2	0,12	0,119335	0,00055549	$3,08569 \cdot 10^{-7}$	
				0,119891		$6,87413 \cdot 10^{-7}$	0,000779

2.1. Calorimetric apparatus

Figures 1 to 3 represent a calorimetric apparatus consisting of a thermostatic container 1 used for realisation of the experiment. A square stove heated by an electric circuit is integrated into the bottom of the thermostatic container. The plate is equipped with a heat exchanger with finned radiator, whose fins face upwards. A square-shaped storage plate made of an insulating material is situated adjacent to the stove. The area of storage plate walls is the same as the area of stove walls. A specimen of the studied material is placed on the storage plate. The proportions of walls of the studied plate are equal to the proportions of other storage plates and the area of the stove. A square storage plate is located adjacent to the studied sample. Probes of temperature difference sensor are inserted in equidistant time intervals at the boundary of areas of storage plates, under and above the studied sample. The sensor is connected to the timer and the spreadsheet processor, which is used for the registration and analysis of measurement results [7]. The interspersing of boards made of conductive and non-conductive material was determined empirically in order to regulate the speed of thermal flow through the system. The goal was to prevent excessively slow or excessively rapid measurement, i.e. to take into account thermal inertia of studied materials. The result of illustrative measurement at the non-stationary heat flow (in the third stage of heat flow) is $\lambda = (0.110 \pm 0.003)$ W/mK. The relative uncertainty of the measurement result is 2.7 %.

The result of illustrative measurement of thermal conductivity λ of the examined building material sample (polyurethane) at the stationary heat flow for the mean material temperature of 67 °C is as follows: $\lambda = (0.1199 \pm 0.0008)$ W/mK. The relative

uncertainty of the measurement result is 0.67 % (Table 1).

The coefficient λ generally depends on both temperature and moisture. For the use of the method, it is especially important that the measured samples are well dried and that their moisture values are mutually comparable, because insulating capabilities of the declared structural material decrease with an increase in moisture. For example, comparative porous-concrete samples of the classes YTONG P2-500 and YTONG P4-500 have the coefficient λ value of 0.12 W/mK in the dried state, while the coefficient λ value would be 0.135 W/mK for the moisture of 4.5 % (according to the ČSN EN 1745 standard). In construction industry, the mentioned material is mostly used for the interior load-bearing walls and interior partitions.

2.2. Measurement results and their interpretation

A new method for measuring physical properties of a new building material is presented in the paper. The measurement was conducted using a newly-designed calorimetric apparatus. The method served to determine material coefficient of a heat insulating material, polyurethane plaster (Table 2).

The measurement concentrated on determination of the thermal conductivity coefficient and the specific heat capacity coefficient, which were defined as follows: $\lambda = (0.110 \pm 0.003)$ W/mK, with the accuracy of 2.7 %, and $c = (809 \pm 20)$ J/kgK, with the accuracy of 2.5 % [8, 9]. Besides weighing and measurement of geometrical proportions, 226 and 223 results of direct partial calculations (Figures 4.a and 4.b, Figure 5) of temperature difference between sample walls, were interpreted and analysed. The database of Excel results is represented by graphs in Figure 4 and Figure 5.

Table 2. Mean material characteristics of comparative and studied specimens as obtained using newly-designed calorimetric apparatus, instrument ISOMET 2114 and indirect measurement

Types of materials Properties of materials	Slabs of porobeton "reference specimen"	Polyurethane mortar (new apparatus)	Polyurethane mortar (ISOMET 2114)
Thermal conductivity, λ	0,12 [W/mK]	0,11 [W/mK]	0,12 [W/mK]
Density, ρ	500 [kg/m ³]	571 [kg/m ³]	-
Specific heat capacity, c	1000 [J/kgK] -	809 [J/kgK] 461 939 [J/m ³ K]	809 [J/kgK] 477 550 [J/m ³ K]
Thermal diffusivity, a	$2,4 \cdot 10^{-7}$ [m ² /s]	$2,38 \cdot 10^{-7}$ [m ² /s]	$2,39 \cdot 10^{-7}$ [m ² /s]

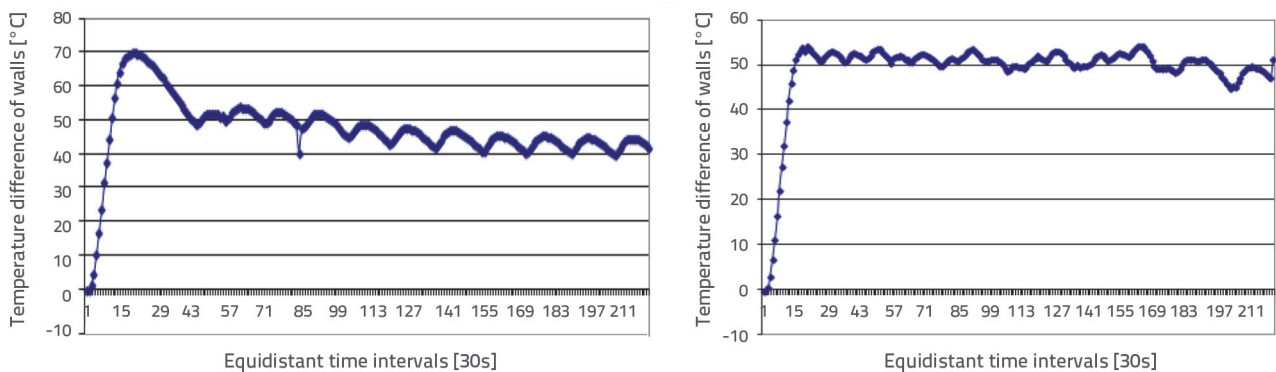


Figure 4. Dependence of temperature difference Δt a) on time in polyurethane specimen 8 cm in thickness; b) on time in ytong specimen 8 cm in thickness

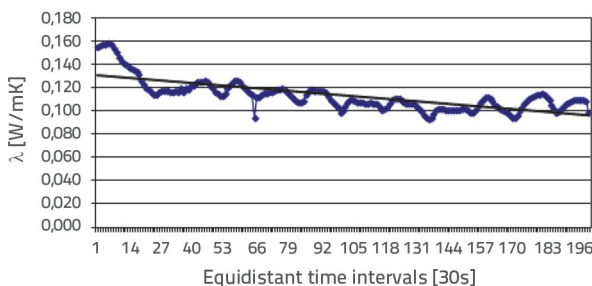


Figure 5. Graphical evaluation of coefficient of thermal conductivity λ in a polyurethane specimen 8 cm in thickness

Measurement results for comparative ytong specimens (Table 2) and the studied specimen made of an insulating material were compared during steady and unsteady thermal flow. Material characteristics are measurable at a steady thermal flow, where the temperature difference between the two localities is kept constant, regardless of thermal capacity of the material. The presented method enables the measurement and analysis of measurement results at a steady thermal flow, but with no condition for keeping constant the temperature difference between board walls.

At the beginning of the specimen bottom heating, the temperature difference between board walls is zero. During steady heating, the temperature difference gradually increases, which leads to an unsteady thermal flow due to the unfulfilled thermal capacity of the specimen. As soon as the thermal flow becomes steady, it takes a relatively short time interval to make the temperature difference constant. Depending on the type of heating, material and geometrical proportions of the specimen, the time interval can be relatively short, and so a "break" in the local extreme takes place. During a longer time interval of the steady thermal flow, it is possible to fit a line, which is parallel with the time axis, with more points. However, if the heating still continues, the thermal capacity of the studied specimen is fulfilled and the specimen then transfers all the received heat to other specimens of the system, and it eventually becomes a heating specimen. The temperature difference between its walls decreases, as its thermal flow switches back to the unsteady one. The experimental specimen cannot be made of

the same material, but it has to be of a comparable one, so that the local extremes of both courses overlap, at least partly. In the intervals of intersections of heating course in time, material coefficients can be determined by calculation Eqs. (5), (6), or graphically (Figure 5), as the mean value of measured data.

Measurement results were compared using the instrument ISOMET 2114. This commercial instrument is a reliable mobile hand-held instrument for direct measurement of the coefficient of thermal conductivity, specific volume heat capacity, coefficient of thermal diffusivity, and temperature (not only for compact but also for bulk and liquid materials) by means of an exchangeable needle and plane probes. Each probe has a built-in memory in which its calibration constants are stored. It is a universal device controlled by microprocessor. As to measurement principle, the device uses a non-stationary contact method of measurement, i.e. the time dependence of temperature response of the studied material to thermal pulses is analysed (thermal flow is induced by electric heating of the resistance heater in the probe that is in direct contact with the tested sample). The curve of temperature as a function of time is sampled at discrete points and, through these samples, regression polynomials are fitted using the least square method. The coefficients of regression polynomials are used in the calculation of thermo-physical parameters. The measurement procedure is relatively simple: at first, the mass and geometric dimensions of the sample are measured, and then the sample is adjusted. The adjustment of the sample is related to the need of ensuring good heat transfer from the probe to the sample and consists in the fact that a smooth area, 0.025 cm in radius, is to be formed. A measuring probe connected to the apparatus itself can then be attached to this area. A several minute lasting measurement process can only then be triggered, and the corresponding results are shown on the display at the end of measurement.

In the case of instrument ISOMET 2114, the resolution capability of direct measurements of physical quantities is quite satisfactory. For the thermal conductivity coefficient measurement it is 0.001 W/mK, for the measurement of specific volume heat capacity it is 103 J/m³K, for the thermal diffusivity measurement it is 109 m²/s, while it is 0.01 °C for temperature measurement. The relative measurement

uncertainty for the coefficient of thermal conductivity is $\pm 10\%$ of the mean value of measurement results ($+ 0.005 \text{ W/mK}$), while it is $\pm 15\%$ of the mean value of measurement results for the measurement of specific volume heat capacity ($+ 3 \cdot 10^3 \text{ J/m}^3\text{K}$). The temperature measurement range of the apparatus is from $0 \text{ }^\circ\text{C}$ to $40 \text{ }^\circ\text{C}$. In the case of the coefficient of thermal conductivity, the measurement range of the apparatus is from 0.015 W/mK to 6 W/mK , in the case of specific volume heat capacity, the range is from $4 \cdot 10^4$ to $4 \cdot 10^6 \text{ J/m}^3\text{K}$, and in the case of sample temperature it is from $- 20 \text{ }^\circ\text{C}$ to $+70 \text{ }^\circ\text{C}$.

Statistical absolute and relative uncertainties of results of partial measurements were evaluated in the course of measurements made by the newly-designed apparatus. To enable a good comparability of measurement results obtained by two different instruments, a database of results was created under comparable conditions (of given samples of polyurethane at the same temperature and the same moisture), and these results were processed using MS Excel.

The coefficient of thermal conductivity of new polyurethane was measured in 10 different localities of the given sample (with $15 \times 15 \times 8 \text{ cm}$ geometric dimensions) using the commercial instrument ISOMET 2114 (Table 2). The measurement result (Table 1) can generally be written as follows:

$$\lambda = \bar{\lambda} \cdot u_\lambda \text{ [W/mK]} \quad (7)$$

λ represents the thermal conductivity coefficient, $\bar{\lambda}$ is the mean value of measurement results, and u_λ is the absolute uncertainty of measurement results [8]. The specific measurement result can be written according to (7) $\lambda = (0.123 \pm 0.002) \text{ W/mK}$. The relative uncertainty of measurement results for the coefficient of thermal conductivity can generally be written as shown in (8):

$$\rho_\lambda = \frac{u_\lambda}{\bar{\lambda}} \cdot 100 \% \quad (8)$$

the measurement result can be expressed specifically according to (8) $\rho_\lambda = 1.6\%$ and evaluated as a relatively accurate result in the scope of laboratory measurements.

Furthermore, the coefficient of specific volume capacity of new polyurethane was measured in 10 different localities of the given sample (with $15 \times 15 \times 8 \text{ cm}$ geometric dimensions) using the commercial instrument ISOMET 2114 (Table 2). The measurement result (Table 1) can generally be written as shown in (9)

$$c_v = \bar{c}_v \pm u_{c_v} \text{ [J/m}^3\text{K]} \quad (9)$$

c_v represents the coefficient of specific volume heat capacity, \bar{c}_v is the mean value of measurement results, and u_{c_v} is the absolute uncertainty of measurement results [8].

The specific measurement result can be written according to (9) $c_v = (477\,550 \pm 5165) \text{ J/m}^3\text{K}$. The relative uncertainty of measurement results for the coefficient of heat volume capacity can generally be written as follows

$$\rho_{c_v} = \frac{u_{c_v}}{\bar{c}_v} \cdot 100 \% \quad (10)$$

the measurement result can be expressed according to (10) $\rho_{c_v} = 1.1\%$ and evaluated as a relatively accurate result in the scope of laboratory measurements.

To enable comparison, the sample density was also measured because ISOMET 2114 evaluated merely the specific volume heat capacity and not the mass. The mean density of all polyurethane samples, $\rho = (571.4 \pm 7.4) \text{ kg/m}^3$, was evaluated at a common room temperature ($24 \text{ }^\circ\text{C}$) and at the same moisture (material of samples was nonhomogeneous, very porous, and thus its density was relatively very low) by indirect measurement of density (by means of precise weighing of polyurethane samples and measurement of geometric dimensions). The mean density of material was measured with a relative uncertainty of measurement results of 1.3% and, in the framework of requirements for the accuracy of laboratory measurements, it can be considered accurate.

In connection with the above-mentioned results, it can be stated that the product of the mean coefficient of specific heat volume capacity of $809 \text{ J/m}^3\text{K}$, and the mean density value of 571 kg/m^3 is $461\,939 \text{ J/m}^3\text{K}$, which corresponds well with the mean specific heat volume capacity measured directly with the commercial instrument ISOMET 2114, which amounts to $477\,550 \text{ J} \cdot \text{m}^{-3} \cdot \text{K}^{-1}$.

The coefficient of specific thermal diffusivity of new polyurethane was measured in 10 different localities of the given sample (with $15 \times 15 \times 8 \text{ cm}$ geometric dimensions) using the commercial instrument ISOMET 2114 (Table 2). The measurement result (Table 2) can generally be written as follows:

$$a = (\bar{a} \pm u_a) \text{ [m}^2/\text{s]} \quad (11)$$

where a represents the coefficient of thermal diffusivity, \bar{a} is the mean value of measurement results, and u_a is the absolute uncertainty of measurement results [8]. The specific measurement result can be written according to (11) as $a = (2.39 \pm 0.06) \cdot 10^{-7} \text{ m}^2/\text{s}$. The relative uncertainty of measurement results for the coefficient of thermal diffusivity can generally be written as follows:

$$\rho_a = \frac{u_a}{\bar{a}} \cdot 100 \% \quad (12)$$

The result of this measurement can specifically be expressed as $\rho_a = 2.5\%$ and evaluated as being relatively sufficiently accurate in the framework of laboratory measurements.

The measurement was carried out on samples of various thicknesses, i.e. both on samples of known material and, comparatively, on samples of the new material. However, the condition for accurate overall measurement results was that both compared samples had to have the same thickness and, simultaneously, the thicknesses were relatively negligible in relation to the remaining geometric dimensions. The most

accurate results were registered for samples with geometric dimensions of 15 x 15 x 8 cm; while the least accurate results were registered for the thicknesses of 6 cm and 8 cm. On the other hand, thermal characteristics evaluation results were highly inaccurate, and hence unusable, for the samples measuring 10 cm in thickness. The base of measurement results described in this paper concerns specifically and representatively specimens measuring 15 x 15 x 8 cm.

Deviations of measurement results were evaluated statistically, i.e. using the uncertainty of type A measurement results, and then using the relative uncertainty of measurement results. The uncertainties of type B measurement results, determined by means of a thermometer, were also considered, namely for temperature difference measurements. Complex type C uncertainties, and consequently their relative uncertainties with regard to the overall measurement result, were evaluated by means of the types A and B uncertainties. For the measurement of specimens with the mentioned thicknesses (from 1 to 8 cm), relative uncertainties of measurement results ranged from 0.5 % to 3 %. Lateral and longitudinal specimen dimensions should not be of a similar order, because a considerable part of the supplied heat escapes through the side walls of the sample; therefore the thickness of 1.5 cm can be recommended, but not 15 cm for the applied square wall specimen measuring 15 cm x 15 cm.

Likewise, measurement results correspond well with tabular values of polyurethane of similar composition. However, the studied material exhibited new structural properties as renovation plaster with very high heat insulating effects. This new plaster fulfils not only the heat insulating function, but also the moisture renovation function. Its additional practical contribution lies in the fact that it does not cause degradation of back-up wall and, simultaneously, re-salinization of masonry due to the change in partial pressure and/or due to the absence of moisture gradient.

All measurements were used as the basis for selection of an optimal formula for new polyurethane materials according to binding requirements for complementary measures in complex renovation activities, i.e. waterproofing, grouting, electro-osmotic methods, etc. In the case of this type of renovation plaster, a polyurethane foam incorporated in the cement-metakaolin matrix makes it possible (as a result of its porosity, i.e. size and structure of pores, and effective modulus of elasticity) to increase by several times the capacity of salt deposition in the renovation plaster structure with heat insulating effects, but without the destruction of this innovative mixture. Such a physical effect is possible because of the influence of increasing the volume of salt crystals and crystallization and recrystallization pressure in the structure of the given mixture connected with it. As a result of these material changes, changes of thermal characteristics (density, thermal conductivity and thermal diffusivity and specific heat capacity) are also predicted. For the purpose of tabulating for practical use, thermal characteristics of

the new material were accurately measured by comparable measurement methods" [1]. The optimal formula from the citation refers to the literature referred in [1]. This is the basic formula (equation) for the PUR foam development [10, 11].

It is satisfactory that new materials exhibit required thermal properties and that they are used widely in construction industry. It is however necessary to implement other subsequent measurements in order to be able to predict their depreciation after long-term use. In addition, basic standard parameters of insulating materials must be respected. The useful life of the studied materials ranges from 30 to 80 years, depending not only on material structure, but also on the inclusion of material in the plaster composition, and on the method of application. For this reason, thermal characteristics of new materials have to be measured accurately, but not only at the initial stage. Measurements have to be subsequently repeated in the long-term at equal time intervals and under appropriate clearly declared conditions. Only after verification of many applications over several decades can it become possible to draw unambiguous conclusions about the prototype material, for the exact specification of which the new apparatus and the new method associated with it were developed [1].

The mentioned device was used to conduct the total of 290 tests on different materials, while 165 tests were performed on different experimental batches of different recipes based on the PUR plaster. The composition of some of them is given in references [1, 2]. Various experimental batches were prepared in order to test thermal properties of polyurethane plaster. The proportion of the PUR component in these batches ranged from 15 to 30 percent by weight.

Various materials can be measured using this method, but always in compliance with conditions specified for its use. The sample measurement from this example is specifically related to measuring properties of building materials with regard to the requirements of individual projects.

Polyurethane foam specimens subject to deterioration by UV radiation have not been applied. The PUR insulating plaster, in which the grains of polyurethane foam are incorporated in the cement matrix at the end of the life cycle, has been applied. In addition, it is expected that the final silicone plaster, resistant to UV radiation, and able to protect the PUR plaster, will be applied onto the polyurethane plaster. Portland Cement CEM I 42.5 R, compliant with EN 197-1 is used as binder. The proportion of various components in percentage by weight: by weight of all components of the experimental batch – PUR 15-30 %, CEM I 42.5 R cement 40-65 %, water 40- 70 %, by weight of the cement – additives 0.5-1.5 %, metakaolin 12-35 %.

The above statement is based on the experimental and applied research, and on practical knowledge: about 90 family houses in the Moravian-Silesian Region of the Czech Republic were heat-insulated with the thermal insulation PUR plaster.

3. Conclusion

Material coefficient measurement results presented in the paper were obtained using a newly-developed method and a newly-designed apparatus. These results were also verified by measurement using the commercial machine ISOMET 2104, and they provide reliable information on stable thermal conductivity in stationary state for a new type of heat insulating plaster, which was analysed in a good insulating and quasi-homogenous environment. In comparison with the traditional apparatus, the main advantage of the new method and apparatus is that it is no longer necessary to keep cooling and heating at a constant temperature. At first, the heat flow through the studied sample is non-stationary but, after some time, the heat capacity of the sample is fulfilled and the thermal flow becomes stationary. The designed testing arrangement can also be applied for testing specific heat capacity, provided that the weight of the tested sample is determined. Having evaluated the sample density, specific heat capacity, thermal conductivity and volume density, we can then use the results for calculating the specific thermal diffusivity coefficient. The above-mentioned advantages, however, require compliance with requirements for comparative measurement of the reference sample. The comparison involves two identical measurement procedures on identical apparatus, the measurement of the comparative sample, and the measurement of the studied sample of material. However, the measurement of reference sample is made one-time only, and the value obtained is saved in the spreadsheet processor

program. Other advantages of the machine are its low manufacturing costs, simple operational maintenance, easy and quick measurement, satisfactory accuracy of results (relatively low uncertainty), and long lifespan of the apparatus, as it does not contain any moving parts.

Modern commercial devices based on non-stationary contact methods measure thermal properties of the material merely on its surface. These methods are based on the assumption that the material is perfectly homogeneous.

The contribution and originality of the new apparatus lie in the fact that thermal coefficients of the material are measured in the entire volume of the tested sample. The new method of measurement [10] is contact-based, comparative, non-stationary, and involves determination of the thermal stationary interval of measurement. It is applicable using a mobile apparatus that is simple to operate. It can also be used in a relatively simple way without installation in the thermostat, and is therefore easier to use when compared to stationary methods with traditional plate-based apparatuses.

Acknowledgements

This paper was supported by the project RMTVC No. CZ.1.05/2.1.00/01.0040, and the IT4 Innovations Centre of Excellence project, reg. no. CZ.1.05/1.1.00/02.0070, the project Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use, Reg. No. CZ.1.05/2.1.00/03.0082 and SGS project SP2014/114.

REFERENCES

- [1] Vaclavik, V., Dvorsky, T., Dirner, V., Daxner, J., Stastny, M.: Polyurethane foam as aggregate for thermal insulating mortars and lightweight concrete. *Tehnički vjesnik – Technical Gazette*. Volume 19, Issue 3, pp. 665-672, 2012.
- [2] Vaclavik, V., Valicek, J., Dvorsky, T., Hryniewicz, T., Rokosz, M., Harnicarova, M., Kusnerova, M., Daxner, J., Bendova, M.: A method of utilization of polyurethane after the end of its life cycle. *Rocznik Ochrony Środowiska*. Volume 14, pp. 96-106, 2012.
- [3] Tsukinovsky, D.: *Study of thermodynamic properties of polyurethane under weak impact conditions*. Ben-Gurion University of the Negev, 1997.
- [4] Ziman, J. M.: *Principles of the theory of solids*. Cambridge, Univ. Press, 1972.
- [5] Bailyn, M.: *Survey of thermodynamics*. American Institute of Physics, New York, 1994.
- [6] VSB-TU OSTRAVA. *The device for identifying the physical properties of solid materials*. Inventors: Kusnerova, M., Valicek, J., Vaclavik, V., Daxner, J., Praha: Industrial Property Office ČR: UV 23861, U1, G01N 25/18 (2006.01), G01N 27/18 (2006.01), Owner: VSB-TUO, Ostrava, CZ a D&DAXNER Technology, s.r.o, Ostrava, CZ, 2012.
- [7] Kostial, P., Ruziak, I., Jonsta, Z., et al.: Experimental method for complex thermo-mechanical material analysis. *International Journal of Thermophysics*. Volume: 31, Issue: 3, pp. 630-636, 2010. DOI: 10.1007/s10765-010-0745-5.
- [8] Kusnerova, M., Valicek, J., Harnicarova, M., et al.: A proposal for Simplifying the Method of Evaluation of Uncertainties in Measurement Results. *Measurement Science Review*. Volume: 13, Issue: 1, pp. 1-6, 2013. DOI: 10.2478/msr-2013-0007.
- [9] Bezjak, M.: Measurement uncertainty associated with the thermal conductivity of building materials, GRADEVINAR 56 (2004) 5, pp. 289-293 (in Croatian)
- [10] VSB-TU Ostrava. Equipment for the identification of physical properties of solid materials. Inventors: Kusnerova, M., Valicek, J., Vaclavik, V., Daxner, J. *Utility model CZ 23861 U1*. 24. 05. 2012, Czech Republic, 2012.