

# Model for Cuboid Shape Samples and its Analysis Used for Measurements of Thermophysical Properties of Sandstone

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A new model for the sample of square cross section with cuboid geometry including the effect of heat loss from the surface of the sample was tested using the theory of sensitivity coefficients. Theoretical calculation of model uncertainty and derived analytical formulas are presented. Results of the uncertainty analysis set out the range of experimental conditions under which the model is valid and the uncertainty of estimated parameters is low. Propagation of error for non-stochastic dynamic measurements based on the sensitivity coefficients shows limitations related to a range of model validity. The analysis improves the accuracy of measurements. The model was used for data evaluation of thermophysical parameters measured on the sandstone from the locality Pravčická brána. Evaluation procedure was tested on experimental data measured under different experimental conditions. In conclusion, the discussion of the experiment optimization is presented.

**Keywords:** Pulse transient method, sensitivity coefficient analysis, thermophysical properties

## 1. INTRODUCTION

**T**HERMOPHYSICAL PARAMETERS (e.g., thermal diffusivity, specific heat and thermal conductivity) reflect the thermodynamical state of the material structure. This state changes with the consideration of the atomic structure arrangement, arrangement of crystalline components and consideration on material defects created in time that are responsible for further development of structure change or degradation. Thermodynamical state should be a measure of the quality, stability and durability of material in regard to industrial use in civil construction.

Measurement of thermophysical parameters of materials in connection with their thermodynamic state reflects various physical and chemical processes and processes in technology like phase changes, domain structure changes, structure relaxation, degradation (deterioration) of materials, ageing, polymerization, vulcanization, sintering of ceramics, curing, sorption/desorption... In principle, the method is applicable in the investigation of degradation of any kind of material. The process of material deterioration of stones or their state and quality comparing its basic or initial state should be monitored by continuous measurements (monitoring) of thermophysical properties too. Pulse Transient Method can be used for the quality control in material testing.

The problems connected with deficiency in a large amount of testing material cause some problems in data evaluation, as an ideal model usually assumes infinitively large specimen. Usually, the shapes of specimens used for measurement are of cylindrical or cuboid form. The finite geometry of the specimen cause additional effects that harm the efficiency and accuracy of the measurement. The contributions to uncertainty come from additional effects caused by differences in ideal and real sample when specimen geometry is limited. The main effects are the heat losses from the sample surface (the heat transfer from the

sample surface to the surroundings); the heat capacity of the heat source and technically the heat pulse is not ideal Dirac function but a pulse of limited duration.

In this paper we discuss the given problem of heat losses for cuboid shape samples.

## 2. SUBJECT & METHODS

### *Principle of the Pulse Transient Method*

Pulse Transient Method belongs to a group of dynamic methods for measurement of thermophysical parameters. The principle is simple. The planar heat source is generating the heat pulse. A temperature response to the heat pulse is recorded by thermocouple placed separately from the heat source (Fig. 1).

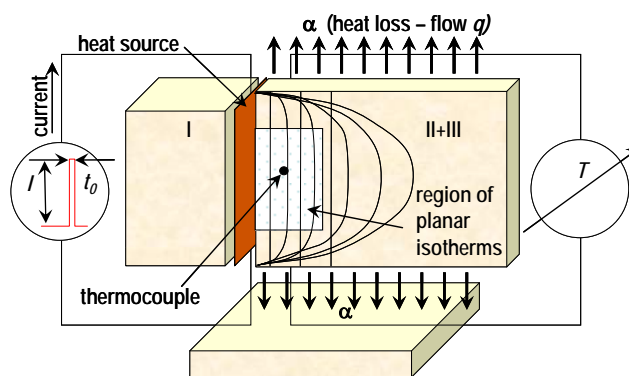


Fig.1. Wiring diagram and the sample set. In between first and second part of the sample set a planar heat source is inserted. The thermocouple for the measurement of temperature response to the heat pulse is inserted in between second and third part.

### *Ideal model*

The temperature response is described by function ( 1 ) :

$$T(h, t) = \frac{2 \cdot q}{c \rho \sqrt{\kappa}} \left[ \sqrt{t} \cdot i\Phi^* \left( \frac{h}{2\sqrt{\kappa t}} \right) - \sqrt{t-t_0} \cdot i\Phi^* \left( \frac{h}{2\sqrt{\kappa(t-t_0)}} \right) \right] \quad (1)$$

where  $i\Phi^*(x) = \frac{e^{-x^2}}{\sqrt{\pi}} - x \cdot \Phi^*(x)$ ,  $T$  is temperature,  $h$  is

thickness of the specimen.  $\Phi^*(x)$  is complementary error function,  $t$  is time. Simple formulas for evaluation based on maximum of the temperature response have been derived. This is called one point evaluation procedure.

$$\kappa = h^2 / 2t_m \cdot f_\kappa, \quad (2)$$

$$c = q / (\sqrt{2\pi e} \rho h T_m) \cdot f_c \quad (3)$$

and

$$\lambda = \kappa \cdot c \cdot \rho \quad (4)$$

where the correction factors  $f_k$  and  $f_c$  were described in [1],  $\lambda$  is thermal conductivity,  $\kappa$  is thermal diffusivity;  $t_m$  is maximum of the temperature response,  $t_0$  is duration of the pulse width,  $c$  is specific heat,  $q$  is heat flux density at source,  $\rho$  is density of material.

#### Model for the cuboid samples

Heat loss effect included in the new model is represented by heat transfer coefficient  $\alpha$  from the sample surface to the surroundings. The problem starts when the planar isotherm of heat front is deformed at the thermocouple region. In the sample cut at Fig.1 a problem is illustrated where this effect causes deformation of planar isotherms. It is evident for specimens having bigger thickness or for the temperature response measured for longer times. Data that are measured within the marked white area in Fig.1 are still able to be evaluated by the ideal model. For the case when thermal isotherms are deformed by this effect we need to introduce a new model. To solve this problem a new model for cuboid geometry was derived accounting the heat transfer coefficient  $\alpha$ .

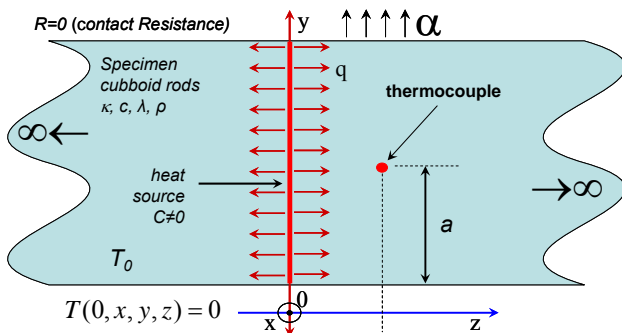


Fig.2. Initial and boundary conditions for the model.

The solution of basic heat equation for initial and boundary conditions that are drawn in Fig.2 is the following temperature response:

$$T(t, x, y, z) = T_0 \frac{w}{2} \sum_{n=1}^{\infty} \sum_{m=1}^{\infty} \frac{b_n b_m}{v_{nm}} F(u, v_{nm}) \varphi_n \left( \frac{x}{a} \right) \varphi_m \left( \frac{y}{a} \right) \quad (5)$$

The variables are

$$\varphi_n(s) = \sqrt{\frac{2\beta}{\beta + \sin^2 \mu_n}} \cos(\mu_n s), \quad T_0 = \frac{qa}{\lambda}, \quad \beta = \frac{a\alpha}{\lambda},$$

$$F_n(u, v) = e^{-2uv} \Phi^*(u-v) - e^{2uv} \Phi^*(u+v), \quad u = \frac{z}{2\sqrt{\kappa t}},$$

$$w = \frac{\sqrt{\kappa t}}{a}, \quad v_{nm} = w \sqrt{\mu_n^2 + \mu_m^2}, \quad b_n = \varphi_n(0) \frac{\sin(\mu_n)}{\mu_n} \text{ and } z$$

axial space coordinate,  $x, y$  transversal space coordinates,  $2a$  transversal size of the sample,  $\alpha$  heat transfer coefficient for sample–ambient interface,  $\mu_n$  are the roots of equation  $\beta \cos \mu - \mu \sin \mu = 0$ .

#### Accuracy estimation

Generally, uncertainty arises from different sources and includes errors in the data measurements, parameter estimation procedure and model structures. Uncertainty analysis calculates how these errors are propagated through the model and evaluate their relative importance which is quantified via sensitivity analysis. This type of uncertainty should be supposed as systematic error. The analysis uses sensitivity data generated by the model, along with the underlying data covariance to assess the degree of similarity (linear dependence) between sensitivity coefficients in the model. If the sensitivity coefficients are linearly dependent on each other, the parameters should not be estimated unambiguously and thus their uncertainty is high.

The analysis was developed in respect to the experimental data set  $\{t_n, T_n\}_{n=1}^N$ , where  $N$  is the number of independent measurements. Model temperature function (5)  $T_{model} = f(t, a, b)$  represents temperature response in time that depends on constants  $b = \{b_j\}_{j=1}^{N_b}$  in model that are determined by different independent measurements and dependent random variables represented by a set of free parameters  $a = \{a_i\}_{i=1}^{N_a}$  that are evaluated by fitting procedure. We used least square optimization to search for  $\min \left\{ \sum_{n=1}^N [T_n - f_n(a, b)]^2 \right\}$ , where  $f_n(a, b) = f(t_n, a, b)$ ,  $t_n$  are deterministic parameters,  $b, \{T_n\}_{n=1}^N$  are independent random variables. The least square optimization gives a system of non-linear equations that is based on sensitivity coefficients [2], (Fig.3.).

$$\sum_{n=1}^N (T_n - f_n) \frac{\partial f_n}{\partial a_i} = 0, \quad i = 1, 2, \dots, N_a \quad (6)$$

Assuming

$$\langle (b_j - \langle b_j \rangle)(b_k - \langle b_k \rangle) \rangle \gg \delta_{jk} u(b_j)^2 \quad (7)$$

$$\langle T_n \rangle \gg f_n(\langle a \rangle, \langle b \rangle) \quad (8)$$

$$\langle (T_n - \langle T_n \rangle)(T_m - \langle T_m \rangle) \rangle \gg \delta_{nm} u(T)^2 \quad (9)$$

$$\langle (T_n - \langle T_n \rangle)(b_k - \langle b_k \rangle) \rangle \gg 0 \quad (10)$$

The expressions in brackets  $\langle \bullet \rangle$  represent the statistical mean values. In the next equation, we estimate the uncertainty

$$u(a_i)^2 \sim \langle (a_i - \langle a_i \rangle)^2 \rangle = \langle (\Delta a_i)^2 \rangle \sim \langle (da_i)^2 \rangle \quad (11)$$

Differentiating equation (6) we obtain a system of equations

$$\sum_{i=1}^{N_a} A_{ki} da_i = \sum_{n=1}^N dT_n \frac{\partial f_n}{\partial a_k} + \sum_{j=1}^{N_b} B_{kj} db_j, \quad k = 1, 2, \dots, N_a \quad (12)$$

Where

$$A_{ki} = \sum_{n=1}^N \left[ (f_n - T_n) \frac{\partial^2 f_n}{\partial a_k \partial a_i} + \frac{\partial f_n}{\partial a_k} \frac{\partial f_n}{\partial a_i} \right] \cong \sum_{n=1}^N \frac{\partial f_n}{\partial a_k} \frac{\partial f_n}{\partial a_i} \quad (13)$$

$$B_{kj} = \sum_{n=1}^N \left[ (f_n - T_n) \frac{\partial^2 f_n}{\partial a_k \partial b_j} + \frac{\partial f_n}{\partial a_k} \frac{\partial f_n}{\partial b_j} \right] \cong - \sum_{n=1}^N \frac{\partial f_n}{\partial a_k} \frac{\partial f_n}{\partial b_j} \quad (14)$$

Now we can estimate uncertainty contribution of any particular measurement of involved parameters like T or b. Solution of equations (12) has the form

$$da_k = \sum_{i=1}^{N_a} A_{ki}^{-1} \left( \sum_{n=1}^N dT_n \frac{\partial f_n}{\partial a_i} + \sum_{j=1}^{N_b} B_{ij} db_j \right), \quad k = 1, 2, \dots, N_a \quad (15)$$

$$u^2(a_k) = \sum_{i=1}^{N_a} \sum_{i'=1}^{N_a} A_{ki}^{-1} A_{ki'}^{-1} \left[ A_{i'i} u^2(T) + \sum_{j=1}^{N_b} B_{ij} B_{i'j} u^2(b_j) \right] \quad (16)$$

$$u^2(a_k) = C_{kT}^2 u^2(T) + \sum_{j=1}^{N_b} C_{kj}^2 u^2(b_j) \quad (17)$$

where the contributions of components from variables and constants are

$$C_{kT} = \sqrt{A_{kk}^{-1}} \quad (18)$$

and

$$C_{kj} = \sum_{i=1}^{N_a} A_{ki}^{-1} B_{ij} \quad (19)$$

the elements of matrix  $\mathbf{A}$  and  $\mathbf{B}$  are defined with equations (13) and (14). We can see that  $\mathbf{A} \sim \mathbf{N}$ , and  $\mathbf{B} \sim \mathbf{N}$ , therefore

$C_{kT} \sim \frac{1}{\sqrt{N}}$  and  $C_{kj}$  is  $N$ -independent. For power-like dependence it is useful to define indices

$$v_{kj} = \frac{\partial \log(a_k)}{\partial \log(b_j)} = \frac{b_j}{a_k} C_{kj} \quad (20)$$

Then for relative uncertainties we can write the equation

$$u_r(a_k)^2 = C_{kT}^2 \frac{u(T)^2}{a_k^2} + \sum_{j=1}^{N_b} v_{kj}^2 u_r(b_j)^2 \quad (21)$$

The normalized sensitivity coefficients were calculated for the same values of thermophysical parameters as those measured in the experimental part and they are given in Fig.3. We can see that optimized region for data evaluation is up to value of  $2F$  dimensionless time.

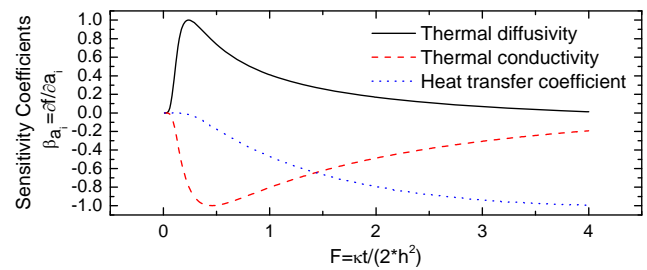


Fig.3. Normalized sensitivity coefficients  $\beta_{a_i} = \partial f / \partial a_i$  (6) derived from temperature function (5) where  $a_i$  denotes free parameter in model, i.e. thermal diffusivity, thermal conductivity and heat transfer coefficient.

### 3. RESULTS

Experimental data were measured in RTB1.02 chamber with temperature stability of 0.01 K. Data from the samples tested under the air as well as vacuum atmosphere were obtained in the temperature range from -22 to 70°C. For the statistic evaluation at least 5 measurements were used to plot Fig.4. Temperature responses were measured for the duration of the heat pulse (heat pulse width) of 3 and 6 seconds. The total time for the recording of the temperature response is derived from the heat pulse width and is 30-times of pulse duration. For the data evaluation two temperature models were used. The first one, considering final pulse width and evaluation procedure based on formulas (2), (3), (4) is derived from the maximum of the temperature response published in several papers [3], [4]. The second one is using fitting procedure by model for cuboid samples having squared cross section and assuming heat losses from the sample surface (5).

The sandstone specimen set was carved in a form of cuboids having finite length. The dimensions of parts I and III were 50x50x30 while part II dimensions were 50x50x10mm. The volume density was 1738.7 kg m<sup>-3</sup>. Porosity measured by weighting dry and water saturated specimen was calculated to 27.5%. Planar heat source was etched of Ni foil and insulated by kapton foil.

Theoretical analysis of model uncertainty (Fig.6) was calculated for temperature response that was generated theoretically for exactly the same values of thermophysical parameters as those of measured sandstone at 25 °C. Data in Fig.6 were calculated for time interval with fixed number of

points involved into evaluation, variable time step and fixed time window that start at  $t=0$ sec. Each data point represents different length of the time interval but the number of points in each interval is the same to preserve the statistical weight of the results.

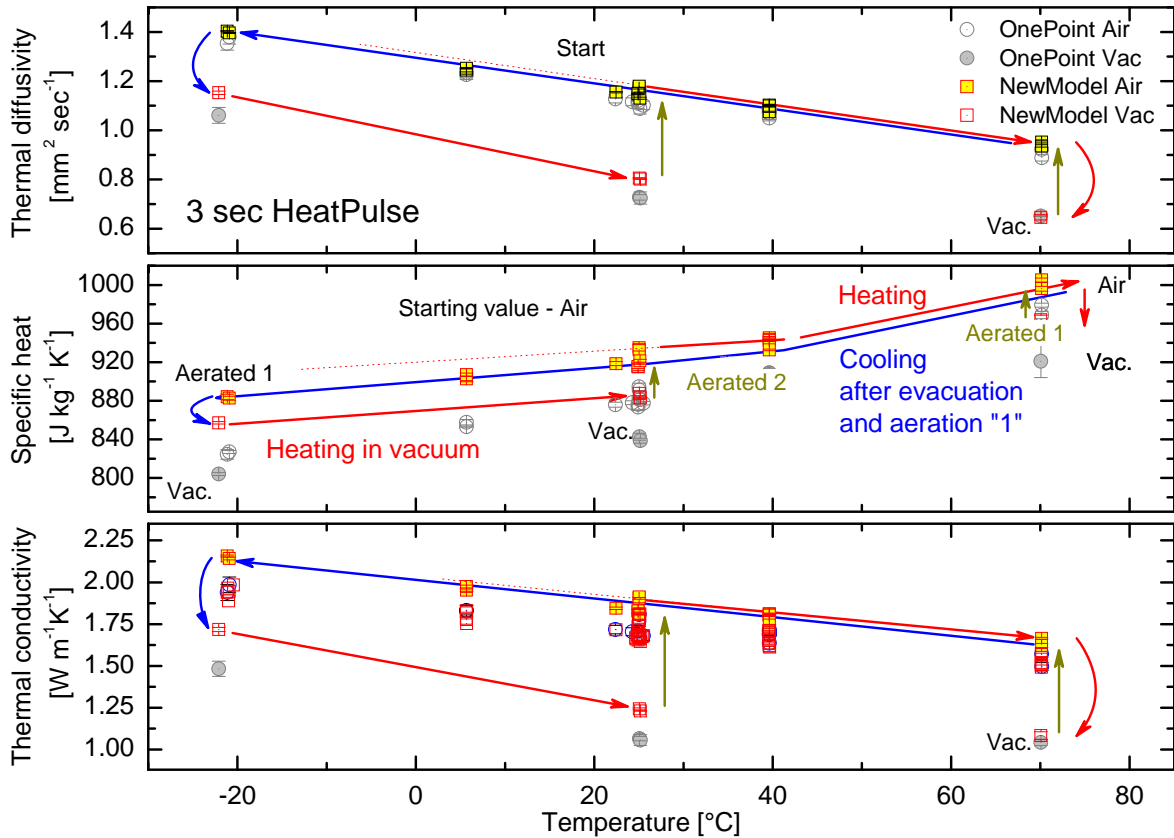


Fig.4. Thermophysical parameters estimated by model fitting and one point procedure in air atmosphere and vacuum. The temperature history as well as vacuum treatment follows the arrows denoting heating and cooling regime. In principle, the temperature dependency of data measured under vacuum follow the measurements performed under the air atmosphere with some shift.

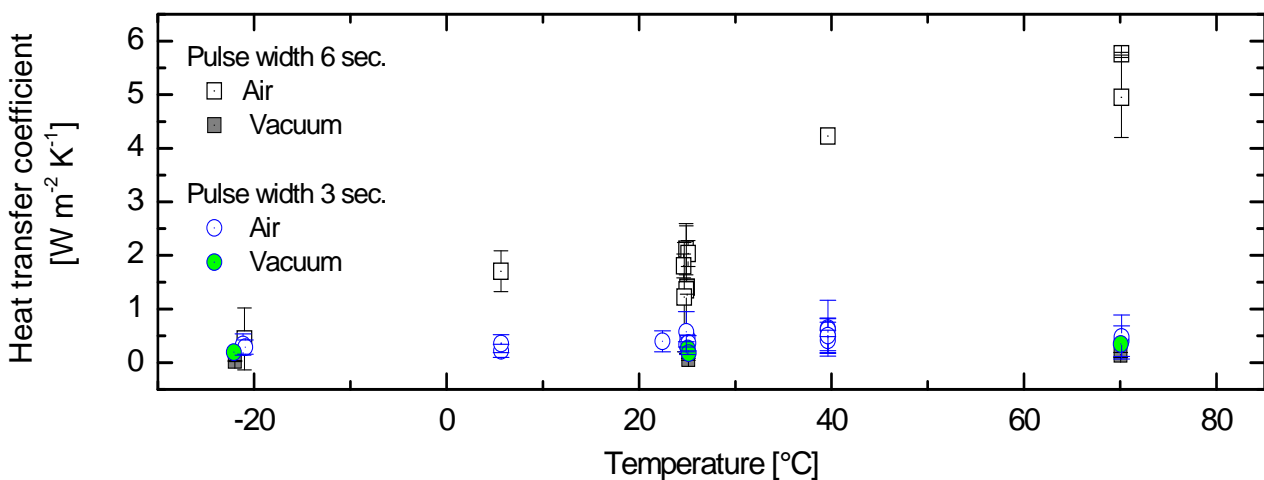


Fig.5. Heat transfer coefficients calculated by fitting procedure for data measured at 3 and 6 seconds of pulse duration as well as for air and vacuum conditions. The low values are those for 3 seconds of pulse duration as well as for data measured under the vacuum. This is the consequence of sensitivity for this parameter. Sensitivity of  $\alpha$  has reasonable values only for bigger thicknesses of material or for longer times of measurement of temperature response. Thus the positive temperature dependence of this parameter is evident only for data measured for 6 seconds of pulse width.

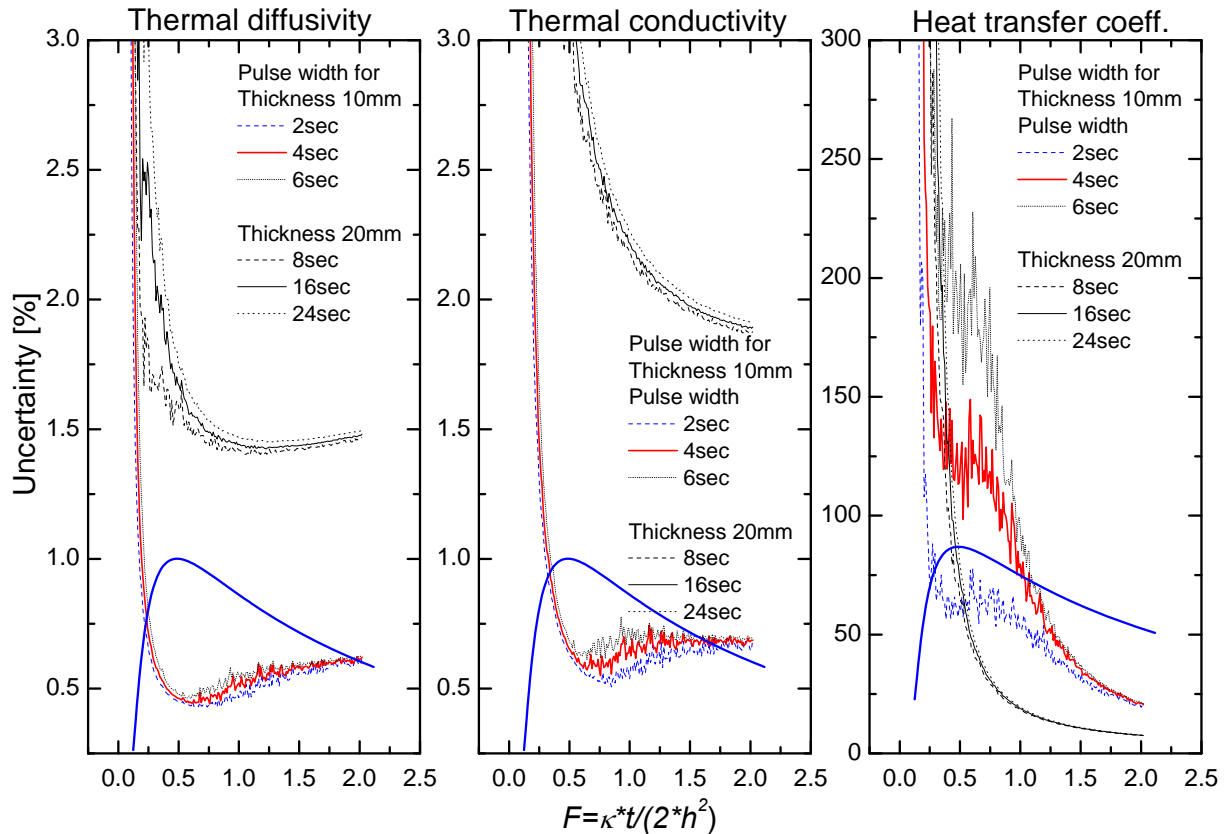


Fig.6. Uncertainty analysis for sample thickness of 10 mm at 2, 4 and 6 seconds of pulse width and thickness 20 mm for 8, 16 and 24 seconds of pulse duration. For the illustration of time relation the maximum of temperature response drawn in blue solid line is at 0.5 of  $F$  - the dimensionless time (Fourier number), that corresponds to 42 seconds in real time. Errors from particular parameters are propagated through the model and their relative importance is evaluated as uncertainty in percent for current time according to the measurement of temperature response.

#### 4. DISCUSSION

Fig.6 illustrates the uncertainties in relation with the total time of the measurement as well as the thickness of the sample. It is an explanation for data plotted in Fig.5, particularly answering the question why the heat transfer coefficient can be evaluated unambiguously only for data acquired for longer times of measurement. Fig.6 illustrates the discussion on the optimal total time for the measurement of transient record. The 6 sec. pulse width corresponds to longer total time of the transient record that finishes at the value of 1.7  $F$  corresponding to 180 sec. The total time of temperature response for 3 sec of pulse duration takes just about 90 sec that correspond to the value of about 1  $F$ . It starts to influence the measurement recorded for longer times or at higher thicknesses of material. We have to wait for a longer time while the heat that penetrates into the material from the heat source and reaches the thermocouple is influenced by heat losses from the surface. The reason is that the sensitivity for this parameter is higher for longer times of measurement and thus the uncertainty is about 5 times lower, which corresponds to lower unambiguity of parameter estimation. Fig.6 shows that the uncertainty of estimation of the heat transfer coefficient is decreasing with increasing thickness of the sample as well as with increasing time of the measurement. In our case for 10 mm sample thickness it is evident that the total time of experimental

recording should exceed 1.75  $F$ . A simpler situation was found with the calculations of uncertainty for thermal diffusivity and thermal conductivity. Uncertainty values are decreasing with increasing measurement time and after getting maximum of the temperature response at 0.5 $F$ , i.e. about 42 seconds in real time axes, it starts to increase slightly. This means that there is no reason for the increase of the measurement time above 1.75  $\div 2F$  (180  $\div$  200 sec.) for this geometry of sandstone material.

#### 5. CONCLUSIONS

The thermophysical properties of sandstone were investigated by Pulse Transient Method for dry state of stone under the air and vacuum conditions. The data were evaluated by two methods – the one point evaluation procedure and by fitting procedure using a new model for cuboid samples that accounts heat losses from the sample surface. The new model shifts up the values of all parameters. The values of thermal diffusivity increased just by small value, but due to data consistency condition the large increase of specific heat causes increase of thermal conductivity values remarkably.

Measured thermophysical parameters of porous sandstone depend on quality of the material and the moisture content in pores. Temperature dependency of the transport parameters (thermal diffusivity and thermal conductivity) is

of negative slope, while the specific heat has positive slope in a given temperature range from -22 to 70°C (Fig.4). The measurements were performed under the air as well as vacuum conditions. Annealing and the vacuum treatment causes lowering of all thermophysical parameters due to the removal of remnant water from pores and structure of the sandstone. The RTB 1.02 chamber should help to avoid problems of insufficient drying because simultaneous evacuation of the chamber and elevation of the temperatures are more efficient in the additional drying process. Differences in all parameters after evacuation and aeration were caused by the additional drying process in vacuum under elevated temperatures.

The uncertainty analysis of the new model of temperature function in respect to the heat transfer coefficient was analyzed and illustrated in Fig.5 and Fig.6. The data illustrate the sensitivity of heat transfer coefficient that represents the heat losses from the sample free surface with regard to the time of the measurement as well as geometry of the specimen. Results show that this parameter is affecting the measurement with increasing time of the measurement and at larger thicknesses of the sample.

The heat transfer coefficient is not possible to estimate unambiguously for shorter times of the temperature response. The heat transfer coefficient from the sample surface to the surroundings is temperature dependent and its values are greater for higher temperatures. The values

measured in vacuum are practically the same over the whole temperature range and also for the data measured for 3 seconds pulse width.

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