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Research article

Utilization of linearization methods for measuring of thermal properties of materials

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Abstract: The aim of the article is to describe the convective cooling of the measured samples and the subsequent processing of the measured to determine the material parameters of the solids in order to develop specialized software. In engineering practice, specifically in the field of materials research, it is necessary to measure the thermal properties of the materials under study. For a variety of materials, which may constitute the substitute for metals, such as steel, alloy, and others, including polymers and composites—especially for the newly developed materials, the table values of these parameters are not available. However, they are important to establish the thermal insulation properties of these materials, for further use in relevant industries. The time constant that determines the rate of cooling of the preheated sample is the basis for determining the additional thermal and technical parameters of the material. Based on the knowledge of the specific heat capacity, after specifying the range of the thermal diffusivity and thermal conductivity of the material, these parameters can be estimated. In order to estimate the coefficients, the non-linear parameter estimation methods can be used, which lead to the iterative calculations. A good candidate for these calculations is the MATLAB program, especially its CurveFitting toolbox. The price of Matlab SW, including CurveFitting Toolbox, is relatively high in terms of using the solution for this purpose. This resulted in the designing of new methods for CurveFitting described in this paper and implemented in the developed program named CurveFit.

Keywords: thermal properties; time constant; determination; linearization methods

1. Introduction

In engineering practice, specifically in terms of materials research, it is necessary to measure the thermal properties of materials. The aim of the article is to describe the convective cooling of the measured samples and the subsequent processing of the measured data to determine the material parameters of the solids to develop a specialized software.

For a variety of materials, which may constitute the substitute for metals such as steel, alloy, and other, including polymers and composites, and especially for the newly developed materials, the tabular values of these parameters are not available. However, these parameters are important for the establishment of, for example, thermal insulation properties of these materials for use in relevant industries [1–3].

The time constant that determines the rate of cooling of the preheated sample is the basis for determining the additional thermal and technical parameters of the material [4–8], such as the heat transfer coefficient and the specific heat capacity that is possible to be used for the determination of thermal diffusivity and thermal conductivity. The prerequisite is the knowledge or at least an estimate of the total heat transfer coefficient from the material into the environment. Moreover, it is necessary to independently determine the density of the sample. When the range of values for the coefficient of heat transfer and heat capacity are entered, the programme determines the estimation of the parameters. Based on the knowledge of the specific heat capacity after setting the range of the thermal diffusivity and thermal conductivity the program estimates these parameters [9–12].

The default function for which it is necessary to determine the parameters is a function in the form $\mathrm{f}(x) = a - b\, e^{-\frac{c}{x}}$. Current methods use the well-known lumped capacity model describing predominantly convective cooling with limited radiation contribution. In order to estimate the coefficients, non-linear parameter estimation methods have been used, which lead to the iterative calculations. A suitable candidate for these calculations is the MATLAB program, especially its CurveFitting toolbox. The high price for solutions, such as MATLAB CurveFitting Toolbox led us to design specific methods implemented in the program CurveFit.

The appropriate transformation function was used and was consequently converted into the linear form of the cooling function. Then the method of the least squares of the linear regression was employed to determine the values for the single-pass determination parameters of the measured cooling process.

The method described in this paper, on the basis of which the application program was created, serves as an evaluation tool for a device that is designed to measure the physical parameters of the materials. By default, the previously established solution was based on MALTAB resources, which due to its cost made the entire apparatus considerably more expensive. Moreover, the advantage of the proposed method leads to the reduction of computational time. In this way the interactive behaviour of the program and manual correction of the selected parameter can be achieved. The response of the program is sufficient even for a large set of input data. Additionally, this method allows rapidly repeated calculations, which greatly streamlines and accelerates the process for assessing multiple samples or repeated measurements for determining the uncertainty of the calculations.

In [13] the problem of the variability effect of the materials' thermal properties when measuring thermal conductivity using steady-state thermal methods is solved. The calculation of the correction, which is originally caused by the problem in the variability of the thermal conductivity of the material, was investigated from the solution where the nonlinear thermal conductivity is calculated for calorimeters operating under steady-state thermal conditions. The condition under which these corrections are zero is obtained.

The approximate analytical solutions for the thermal properties of unconsolidated materials are given in [14]. The relative error of the proposed solution is less than one percent.

An experimental-computational system developed at the Thermal Laboratory, Department Space Systems Engineering, Moscow Aviation Institute (MAI), is presented in [15] based on inverse heat transfer problems. The describing system investigates the materials in conditions of unsteady contact and/or radiation heating over a wide range of temperature changes and heating rates in a vacuum, air and inert gas medium.

Experimental determination of the effective thermal capacity function and other thermal properties for various phase change materials using the thermal delay method is presented in [16]. The presented thermal delay method is an improved version of the well-known T-history method, which is widely used for thermal properties measurement of phase change materials (PCM).

A significant number of measurements of the present study include the thermal properties of various practically interesting PCM: (a) the temperatures at the ends of the two-phase region; (b) the liquid and solid PCM thermal capacities; (c) the phase change heat; (d) the heat storage capacity during any specified temperature range; and (e) the effective thermal capacity function, which is a very important and useful property for practical applications.

The paper [17] introduces the basic concepts of the parameters' measurement and discusses the heat transfer in solids. The attention is paid to methods utilizing no stationary temperature fields, especially significant are the photo thermal methods in which the temperature disturbance in the investigated sample is generated through light absorption. It is shown that by using these techniques it is possible to determine the thermal diffusivity of a wide range of samples.

The results of the thermal diffusivity investigation of the ground in the polar region, which is based on the propagation analysis of the thermal wave generated by sun-light, are also presented. Based on the chosen examples one can state that photo thermal techniques can be used for the determination of the thermal properties of very different materials.

Determination of thermal properties of cross-linked EVA (Ethylene Vinyl Acetate) encapsulate material in outdoor exposure by TSC (Thermally Stimulated Current) and DSC (Differential Scanning Calorimetry) methods is presented in [18]. In this article the thermal properties of unaged and aged EVA encapsulate material were measured by thermal analysis methods, such as TSC and DSC.

A flash method of measuring the thermal diffusivity, heat capacity, and thermal conductivity is described for the first time. A high-intensity and short-duration light pulse is absorbed in the front surface of a thermally insulated specimen, which is a few millimetres thick and coated with camphor black. The data of the resulting temperature of the rear surface is obtained via thermocouple and recorded with an oscilloscope and camera [19].

The complex description of the heat transfer in the material is given in the book Conduction of Heat in Solids [20]. This book describes the known solutions to the problems of the heat flow with a detailed discussion on the most important boundary value issues.

Based on the above mentioned articles, the methods for measurement of thermal properties of materials can be clearly seen as complicated and need the use of precise and sophisticated apparatuses. In this article, the proposed methods use a simple apparatus. The method is based on cooling of the preheated material. The MATLAB CurveFit toolbox was used for the analysis of measured data. This solution was too expensive and therefore a special program was built to retrieve the measured data from the infrared probe and for consequent data analysis.

2. Materials and methods

This section discusses the basic principles of the linearization method and its implementation in the user program.

2.1. The process of cooling material

The principle of the search for parameters of the material is based on finding the equation coefficients that describe the trajectory of the cooling material.

It is clear that the conditions of the heat transfer from a cooling body may not in general be determined solely by convective heat transfer from the surface of heat exchange area S_c into the surroundings. There is also simultaneous demonstration of radiation heat transfer through the surface of the heat exchange area S_r.

If the body has internal sources of heat with a total output of W_g and, at the same time, a flow of heat with a density of q_s is supplied or discharged through some part of its surface S_s (e.g. by conduction due to contact with other bodies), the differential equation of energy balance of the "thermally thin" body has the form of

$$\frac{\rho V c_p \mathrm{d}T(t)}{dt} = \pm q_s S_s - h_c S_c[T(t) - T_\infty] - \varepsilon\rho S_r[T(t)^4 - T_\infty^4] \pm W_g \tag{1}$$

where ε is the emissivity coefficient of the area surface with radiation heat transfer, while σ is the Stefan-Boltzmann constant.

If there is an evidence of radiation heat transfer and provided that

$$\mathrm{q_s} = 0, \mathrm{W_s} = 0 \text{ and } \mathrm{S_c} = \mathrm{S_r} = \mathrm{S}, \tag{2}$$

Equation 1 acquires the form of

$$\rho c_p L \frac{dT(t)}{dt} = -h_c[T(t) - T_\infty] - \varepsilon\rho[T(t)^4 - T_\infty^4]. \quad (3)$$

If the radiation heat flow density is expressed using the coefficient of radiation heat transfer h_r in the form of

$$\varepsilon\sigma\{T(t)^4 - T_\infty^4\} = h_r[T(t) - T_\infty], \quad (4)$$

then

$$h_r = \varepsilon\sigma[T(t) + T_\infty]\{T(t)^2 - T_\infty^2\} \quad (5)$$

and after adjustments, the following is acquired

$$\rho c_p L \frac{dT(t)}{dt} = -h_t[T(t) - T_\infty]. \quad (6)$$

where

$$h_t = h_c + h_r \quad (7)$$

represents the heat-dependent total, or the so called combined heat transfer coefficient through convection with the coefficient of h_c and radiation with the coefficient of h_r.

For a heat flow from a sample to the environment with temperature T_∞ Newton's cooling law can be written in the form

$$\frac{dQ}{dt} = -h_t(T - T_\infty), \quad (8)$$

where Q is the heat given to a sample, h_t is the total heat transfer coefficient of a sample, S is the total heat flow area, T_∞ is the environment temperature.

For the lumped capacitance method solution of surface temperature T can be found from the differential equation

$$m\, c_p \frac{dT}{dt} = -h_t(T - T_\infty), \quad (9)$$

further

$$m = \rho\, V = \rho\, S\, L, \quad (10)$$

where ρ is density of the sample, L is sample thickness and S is the effectively cooled surface.

In this case the sample is cooled on both sides and it can be written as

$$S^* = S_{top} + S_{lower} = 2S. \quad (11)$$

After integration of Eq 2 the following is obtained

$$\left[\rho\, S\, L\, c_p \ln(T - T_\infty)\right]_0^t = \left[-2\, h\, S\, t\right]_0^t. \tag{12}$$

If the following is set

$$\tau = \frac{\rho\, L\, c_p}{2\, h_t}, \tag{13}$$

then

$$\left[\ln(T - T_\infty)\right]_0^t = -\frac{t}{\tau} \tag{14}$$

and finally, for the master function one can write

$$T = T_\infty + (T_0 - T_\infty)\, e^{-\frac{t}{\tau}}. \tag{15}$$

Relation among k, c_p and α has the form

$$k = \alpha\, \rho\, c_p, \tag{16}$$

where k is sample thermal conductivity, α is diffusivity, c_p, is specific heat capacity and ρ is the sample density.

Validity of the model is verified by the Biot number Bi in the form

$$Bi = \frac{h_t L}{k} \ll 1, \tag{17}$$

where

$$h_t = h_c + h_r. \tag{18}$$

The analysis of the simulation results showed that the radiation heat transfer coefficient observed at the temperature range of 25–26 ℃ is practically a linear function of the temperature with increasing trend. The temperature range is usually given by the temperature of the laboratory. However, the value of h_r coefficient within the monitored temperature interval does not change by more than 0.5%, which is why, taking into account the approximate 5% model accuracy, it can be approximated by its arithmetic average or median, which is less sensitive to deviation and extreme values.

The comparison shows that even at room temperatures, with relatively very little temperature differences between the cooling body and the surroundings, the value of radiation heat flow density is considerably high. Radiation heat flow in the given case has an average of more than 23.88% share on the total heat discharge from the surface of the body. That is why the description of the cooling process must usually take into account both convective and radiation heat transfer mechanisms, and it must take into account the combined nature of the thermal interactions of the body with the surroundings. This more accurately corresponds to the macroscopic description of the heat exchange processes than during the application of a simple exponential model of the first order which neglects radiation.

The value of the radiation heat transfer coefficient is calculated from the relation

$$h_r = -\varepsilon\,\sigma \left\{\frac{T(t)^4 - T_\infty}{\{T(t) - T_\infty\}}\right\}. \tag{19}$$

With increasing temperature differences between the cooling body and its surroundings, the possibility for approximation of the radiation heat transfer coefficient using a constant value is definitively lost. At higher temperatures and temperature differences, the temperature dependence of the convective heat transfer coefficient starts to be recognisable.

Higher temperatures can also show thermal dependence of other physical parameters concentrated in the relaxation time, thus losing the character of a constant value.

Since Eq 15 is nonlinear, it is necessary to use a general fitting procedure to obtain unknown coefficients T_∞ and τ. These functions are implemented in the environment CurveFit toolbox of the MATLAB.

2.2. Linearization as a substitute for general fit procedure

The idea that the program CurveFit uses is based on the fact that if the known final temperature T_∞, the ambient temperature at an adequate distance from the cooling body to which the body cools down to during a sufficiently long time, then the beginning of the reference coordinate system of the cooling function can be shifted with the final temperature and a modified relationship can be obtained

$$T - T_\infty = (T_0 - T_\infty)e^{-\frac{t}{\tau}} \tag{20}$$

Here, the value $y = T - T_\infty$ asymptotically approaches zero. The relationship can be linearized by the transfer to the logarithmic scale in order to receive

$$\ln(T - T_\infty) = \ln(T_0 - T_\infty) - \frac{t}{\tau} \tag{21}$$

The equation shows the linear relationship between the magnitude $\ln(T - T_\infty)$ and time. To simplify the next calculation equation, it can be modified into the form

$$\ln(T - T_\infty) - \ln(T_0 - T_\infty) = -\frac{t}{\tau} \tag{22}$$

Substituting $y = \ln(T - T_\infty) - \ln(T_0 - T_\infty)$ and $a = -\frac{1}{\tau}$ the equation of the line passing through the origin of the coordinate system is obtained.

$$y = a\,t \tag{23}$$

The expression $a = -\frac{1}{\tau}$ indicates the slope of the line. This slope can be found using the method of least squares.

The dependence of the surface temperature on the body at the time is given as the set of points *S* containing n individual measurements $S_i = [T_i,\ t_i]$ as an ordered pair of values, where T_i indicates the temperature of the surface of the body measured in time t_i. Index *i* takes values from one to *n*.

For easy calculation denote the constant $= (T_0 - T_\infty)$, which can be always calculated and is the natural logarithm of the difference between the initial and the final body temperatures. It can be expected that the end temperature of the body cooled in an adequate time will be equal to the ambient temperature, which is considered during cooling at a constant value and can be therefore identified at the beginning of the measurement.

The method of the least squares slope of the line was used, which started the search for the slope of the line $y = at$ passing through the origin of the coordinate system

$$a = \frac{\sum_{i=1}^{n} t_i\, y_i}{\sum_{i=1}^{n} t_i^2} \tag{24}$$

2.3. Reliability of parameter estimation

The value of the variable y is calculated for each point of the relationship

$$y = \ln(T_i - T_\infty) - b \tag{25}$$

The overall variability of the response variable y_i is expressed as the total sum of the squares (TSS, total sum of squares). It is calculated by summing the differences of the squares between the values y_i and the overall arithmetic average.

$$TSS = \sum_{i=1}^{n} (y_i - \bar{y})^2 \tag{26}$$

The measure of the explained variability (Model Sum of Squares, MSS) is again a variation of the overall average $\bar{y}$, but in this case it means the variability of the fitness values $\hat{y}$ (e.g. for the time t we calculate using the regression coefficients):

$$MSS = \sum_{i=1}^{n} (\hat{y} - \bar{y})^2 \tag{27}$$

Using the model of unexplained variability—the residual sum of squares (RSS, Residual Sum of Squares), it can be calculated as the difference

$$RSS = TSS - MSS = \sum_{i=1}^{n} (y_i - \bar{y})^2 - \sum_{i=1}^{n} (\hat{y} - \bar{y})^2 \tag{28}$$

or directly using the residuals $e_r = \hat{y} - y_i$

$$RSS = \sum_{i=1}^{n} e_r^2 = \sum_{i=1}^{n} (y_i - \hat{y})^2 \tag{29}$$

The coefficient of determination R^2 indicates what portion of the total sum of squared deviations was explained by fitting regression functions. The coefficient of the determination is determined by the relation

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y_i - \hat{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \tag{30}$$

If it would not be necessary to calculate R^2 then a can be calculated directly by singularly passing through all the measured points. For the calculation of R^2 in the first passing through, the value of the variable Sum is calculated and the total number of points of the measured trajectory N are counted. The content of both of these variables is needed to calculate the average $\bar{y}$.

In the second stage the values R^2 are calculated, in which from the estimated parameter it is possible to determine the value of the variable a at the specific moment and gradually enumerate the partial sums RSS_i and TSS_i, where

$$\mathrm{RSS_i} = \mathrm{RSS_{i-1}} + (y_i - \hat{y})^2 \tag{31}$$

and

$$\mathrm{TSS_i} = \mathrm{TSS_{i-1}} + (y_i - \bar{y})^2\,. \tag{32}$$

In order to eliminate the effect of the noise of the measurement evaluation only to those valid values during the cooling, they need to satisfy the condition that the temperature of the sample is greater than a predetermined temperature difference above the ambient temperature.

If the final temperature is known, it is possible to directly determine the slope of the line, as it is described above.

In the case that the target temperature is unknown, the algorithm searches the minimum sum square for different target temperature of cooling. The default value is set up either by ambient temperature or by the default value, which takes the lowest deviations by changing the estimate of the final temperature.

The found time constant will serve for determination of the dependence $c_p, [J.kg^{-1}.K^{-1}]$ and $h\ [W.m^{-2}.K^{-1}]$.

2.4. The algorithm of the fit procedures

When the final temperature is unknown, the algorithm finds it using a method of interval dividing. First the algorithm finds the direction for increasing accuracy of the final temperature. Then it discovers the point in which the accuracy decreases. Between this point and the last point, the algorithm finds the final result that satisfies the required accuracy.

3. Results and discussion

3.1. Comparison of the results using the CurveFitting Toolbox with the proposed solution

Data in the experiments was measured through real physical specimens, no physical simulation was performed. This is also related to the verification of the results, which were performed by comparing with the reference MATLAB software.

Results for the five curves of cooling rates that are determined by both methods are summarized in Table 1.

Table 1. Results given by MATLAB toolbox and program CurveFit.

Specimen	MATLAB				CurveFit			
	Points	Ta (s)	Amb. Temp (℃)	R^2	Points	Tau (s)	Amb. Temp (℃)	R^2
Mn Steel	28000	119.4	23.78	0.9995	28000	120.4	23.28	0.995
PMMA	11520	114.8	26.21	0.9995	11520	112.3	26.28	0.999
HDPE	11507	40.37	22.81	0.9992	11507	44.27	22.68	0.981
Cupper	24000	49.89	25.5	0.9988	24000	51.44	25.26	0.995
Aluminium	57601	122.8	26.81	0.9988	57601	125.8	26.56	0.993

Based on the obtained results it is evident that both methods are in terms of accuracy completely equal. Different results are given by a different algorithm of numerical evaluation.

The paper does not solve the whole process of thermal data determination but only its first part, oriented on determination of relaxation time. Nevertheless, in this state it is possible to combine experimental and table data to obtain the Biot number. Further steps of the software for the other constant determination are under development and they are not presented in this paper.

Based on the linearization method proposed in the previous sections, data about surface temperature for each time is plotted onto the chart on the left half of the form of the program CurveFit without linearization. During the process of the charting, the important characteristics of the cooling down process are determined (the maximal and minimal temperature of the material). These values are used to first determine the time constant for the cooling process. The red line in the chart is the line presenting the linearized process of cooling and the blue line shows the original linearized curve. Both (curve and line) draw the zero point of the coordinate system of the chart. At the same time, the statistical characteristics (coefficient of determination and sum of errors) are computed.

Figure 1 provides a good image of the detected ambient temperature. The left chart (a) shows curves of the ambient temperature being set too high (the real ambient temperature is lower than the one predicted). It can be seen that the trajectory of the linearized measured cooling temperature in the beginning is below the computed ideal linearized trajectory (in the figure it is the red line named Zero Point). On the opposite in the right chart (b) one can see the situation in which the predicted ambient temperature is too low (the ambient temperature is lower than predicted). Then the original measured and linearized curve lies in its beginning below the ideal one.

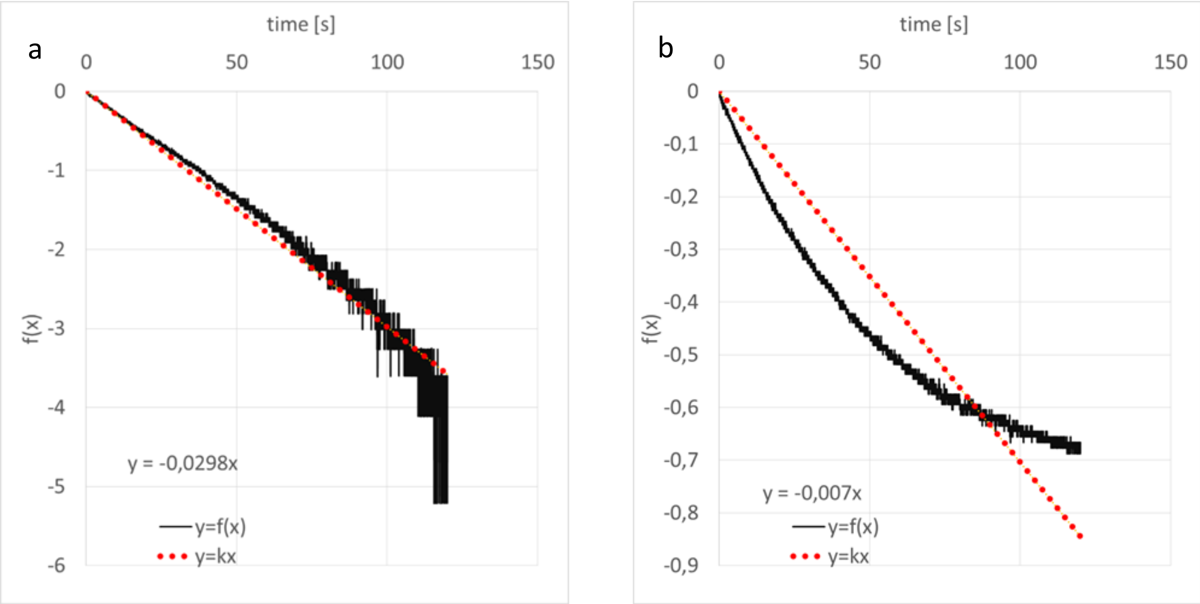

Figure 1. Linearized cooling curve, (a) too high ambient temperature, (b) too low ambient temperature.

In Figure 2 we can see two possibilities of the presentation of given results. In the left chart the linearized cooling trajectory is shown. It is evident that the time constant of cooling changes during the whole time. In the beginning the cooling rate is faster (lower time constant) and in the end of the process the cooling rate is slower (the time constant is higher than at beginning of the cooling). This behaviour of cooling down process is obvious from the linearized trajectory but from the original trajectory in the real coordinate system it may be said that the process and the identifying parameters (time constant and the ambient temperature) are correct.

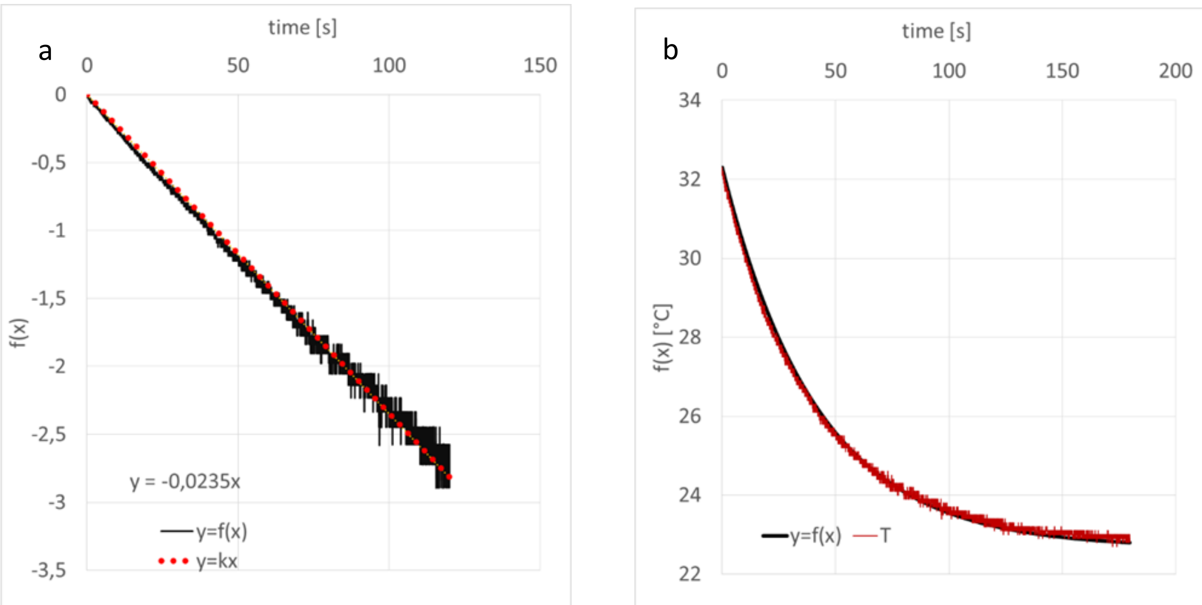

Figure 2. Comparison of results in logarithmic (a) and linear scale (b).

3.2. *The program CurveFit*

The customized fitting procedure is a part of s implemented in a program that allows us to evaluate the thermal properties of materials. The program CurveFit is used for analysing the results of measuring of the materials' thermal properties. The input data are the cooling curves, which are obtained through the non-contact temperature measurement device. Data is provided in the form of a text file with specified parameters.

The program can retrieve this data and display it as a graph (Figure 3). From this set of comprehensive data an appropriate compact area can be selected, which is suitable for subsequent processing. The data is used to estimate parameters of cooling curves, especially for the time constants and the final temperature of the cooling sample.

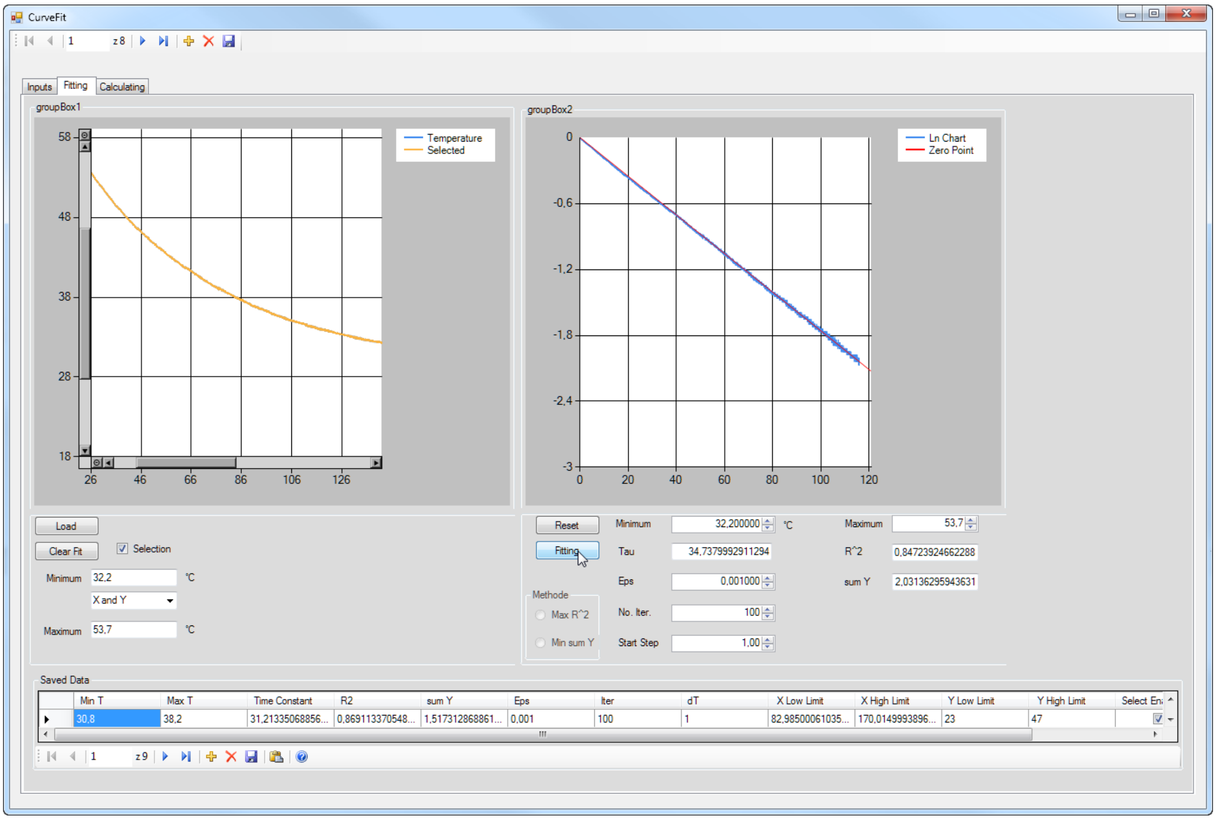

Figure 3. Screenshot of developed SW CurveFit—Constant determination using graph mode.

The time constant is fundamental for determining the additional thermal and physical parameters of the material. The prerequisite is the knowledge or at least an estimation of the general heat transfer coefficient of the material into the surrounding area. From the size of the sample and its mass, the density of the sample material is determined. When the range of values of the heat transfer coefficient and heat capacity are entered, the estimating of these parameters is determined programmatically (Figure 4).

In Figure 4, the computed trends for cooling in the normal coordinates are given. The green curve in the middle (the chart area in the left bottom corner) is the one computed from the predicted time constant and ambient temperature, the other two curves i.e. the red one is calculated from the lower range of the parameters which makes the time constant lower than predicted, while the blue one is calculated from the higher parameters, which on the other hand results in a high time constant. When both red and blue curves lie under or over the correct cooling curve, the computing of the parameters ended without results.

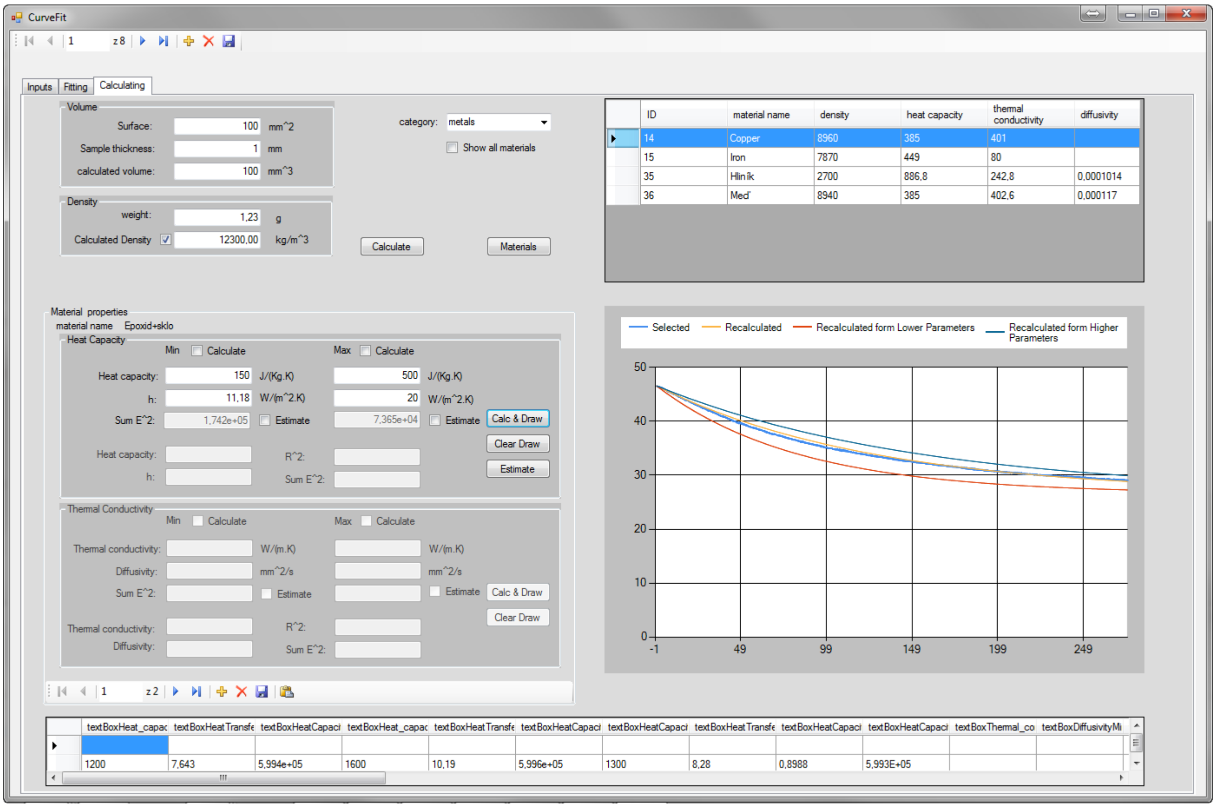

Figure 4. Screenshot of developed SW CurveFit—Constant determination using calculating mode.

Based on knowledge of the specific heat capacity and specifying the range of thermal conductivity and thermal diffusivity the program estimates these parameters.

All data obtained during the work with the program can be saved in a database for later use.

3.3. The benefits of the proposed solution

The used method offers independent determination of specific heat capacity and heat transfer coefficient from one measurement of cooling curve. Previously, it was only possible to use MATLAB software. The main contribution of this paper lies in the creation of the independent CurveFit software, a solution free of MATLAB. The proposed solution has a lower cost in case the standard solution using MATLAB toolbox including CurveFit toolbox is not used.

The developed solution of CurveFit SW is for free, with a USD 0 licence fee. The program runs in a standard Microsoft Windows environment with installed .Net Framework version 4 and higher. SW is simple to use and provides full features with algorithm, which is also implemented in the MATLAB version. DotNet framework has been used for the rapid development of the application, which was verified by the algorithms of evaluation of measured data, as it was described in the theoretical part of this article. The core of the solution can be easily converted into a multiplatform application.

The producer of the CurveFit program is the Department of Automation and Computer Technology in Metallurgy, VSB-TU Ostrava, Czech Republic.

4. Conclusions

This paper shows the possibilities of linearizing the cooling curve of measured material while determining the cooling rate, the time constant and the temperature at the end of the cooling process. These properties can be used to determine other thermal properties of materials, as shown in [9]. This method is very useful for programming realization. The algorithm of computing time constant is one passing algorithm with linear dependency on the number of measured points. The MATLAB toolbox using nonlinear regression is replaced by user program independent of the MATLAB environment.

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Conflict of interest

All authors declare no conflicts of interest in this paper.

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