

## CONTACTLESS METHOD OF SPECIFIC HEAT CAPACITY DETERMINATION

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### ABSTRACT

The paper deals with theoretical and experimental aspects of the application of the lumped capacitance model (LCM) for the study of heat transport in different materials. The patented construction of the measuring chamber together with special software, the fundamental features of which are presented in this paper, enable the contactless evaluation of the specific heat capacity at the constant pressure  $c_p$ . The time constant  $\tau$  that determines the rate of cooling of the preheated sample is the fundamental value for determining of the  $c_p$ . A new method of curve fitting was subsequently developed and implemented into the new software. All theoretical outputs were experimentally tested on a relatively large scale of materials with success.

### INTRODUCTION

In engineering practice, it is necessary to measure the thermal properties of materials. For a variety of materials such as some steels, alloys, polymers and composites, especially for newly developed materials, table values of thermal properties are not available. But these properties are important for the determination of their engineering applications. [1-3].

In [4], the utilization of the extended plane source method (EDPS) for measurements of thermal conductivity using the steady-state thermal regime is described.

The approximate analytical solutions of the thermal properties of unconsolidated materials are provided in [5].

Different methods of the measurement of the thermal properties of solid materials are described in [6-11].

The time constant that determines the rate of cooling of the preheated sample is the basis for determining the specific heat capacity [4-8]. The prerequisite is knowledge or at least an estimate of the total heat transfer coefficient from the material into the environment. It is necessary to independently determine the density of the sample.

In this paper we present the contactless method of specific heat at constant pressure measurement by application of the LCM method and a corresponding software solution.

### THEORY

We can describe the temperature decrease of a preheated solid material cooled in a predominantly convective regime by the lumped capacitance model (LCM) starting from a differential equation [12]

$$mc_p \frac{dT}{dt} = -h_t S(T - T_\infty) \quad (1)$$

$S$  is the effectively cooled surface. In our case, the sample is cooled on both sides of the sample [13]. According to experimental analysis in our case, we can set  $h_t \approx (15-25)$ .

The final solution of equation 1 for temperature decay in the convective cooling regime of a solid sample is in the form

$$T = T_\infty + (T_0 - T_\infty) \exp(-t/\tau) \quad (2)$$

The finding of the time constant will serve to determine the dependence  $c_p$  [ $J \cdot kg^{-1} \cdot K^{-1}$ ] and  $h$  [ $W \cdot m^{-2} \cdot K^{-1}$ ]

By the method of the least squares slope of the line, we are looking for the slope of the line  $y = at$  passing through the origin of the coordinate system [14]

$$a = \frac{\sum_{i=1}^n t_i y_i}{\sum_{i=1}^n t_i^2} \quad (3)$$

We calculate the value of variable  $y_i$  from the following equation

$$y_i = \ln(T_i - T_\infty) - b \quad (4)$$

The coefficient of determination is expressed by the relation

$$R^2 = 1 - \frac{\sum_{i=1}^n (y_i - \bar{y})^2}{\sum_{i=1}^n (y_i - \bar{y})^2} \quad (5)$$

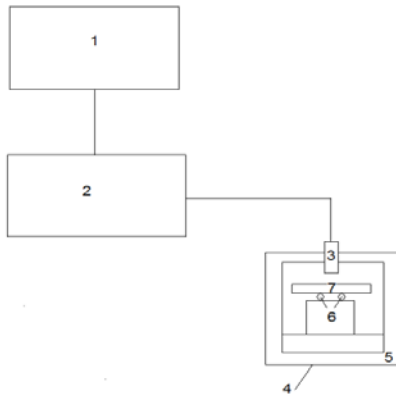
In the case when the target temperature is unknown, the algorithm searches the minimum sum square for the different target temperature of cooling.

The algorithm in the initial step is based on the intersection of the given and possible intervals of both quantities ( $h_t$  and  $c_p$ ). Once the common intervals of meaningful values have been determined, the algorithm tries to find a single value using the interval division method by gradually decreasing the interval of both required values.

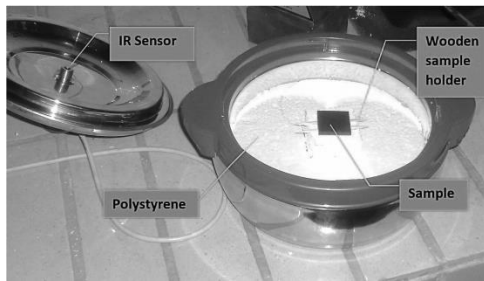
### EXPERIMENTAL EQUIPMENT AND $C_p$ EVALUATION

The equipment consists of a thermally insulated chamber. The cover of the chamber has an opening for a Raytek THERMALERT MID 02 pyro-electric sensor.

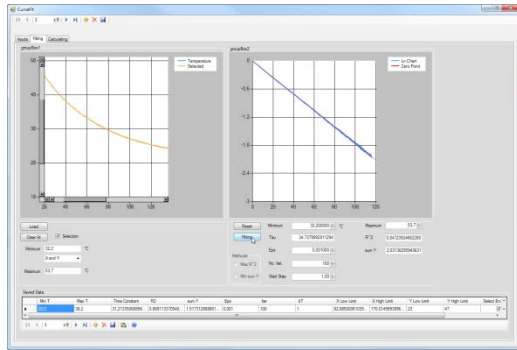
A diagram of the measuring system is in Fig. 1 (details in [15]). Real view of the measuring chamber is in Fig. 2.



**Figure 1** Schema of the apparatus : 1 - PC, 2 - electronic equipment for IR sensor and automatic data acquisition, 3 - IR sensor, 4 - adiabatic chamber, 5 - thermal isolation, 6 - feeble conductive sample support, 7 - sample [15].



**Figure 2** Real view of the measuring chamber



**Figure 3** Printscreen of developed SW CurveFitT

The measured samples must have the dimensions of about  $(10 \times 10 \times 2) \text{ mm}^3$  and must be finely ground. Matt black spray-paint is applied on all sides of the samples in order to ensure they have the same emissivity. The sample is heated above room temperature in a thermostat. After removal from the thermostat, the sample is quickly placed into the measuring chamber which will be closed. It is clear that at the beginning of the sample cooling the transient process take place. In the process of the sample cooling, the relaxation time is not

constant in the whole range. To judge this process, we have to choose a proper interval where  $\tau$  is approximately constant according to the relation

$$\tau = -\frac{t}{\ln \varphi} \quad (6)$$

where

$$\ln \varphi = \ln[(T - T_\infty) - (T_0 - T_\infty)] \quad (7)$$

Relations 6 and 7 enable the setting of a proper, nearly constant interval of  $\tau$ .

**Table 1** Data comparison computed by Matlab software and the CurvefitT one for different materials

		Matlab		
Specimen	Points	$\tau$ [s]	Amb. temp.	$R^2$
Mn steel	28000	119.4	23.78	0.9995
PMMA	11520	114.8	26.21	0.9995
HDPE	11507	40.37	22.81	0.9992
Copper	24000	49.89	25.5	0.9988
Aluminium	57601	122.8	26.81	0.9988
		CurveFitT		
Specimen	Points	$\tau$ [s]	Amb. temp.	$R^2$
Mn steel	28000	120.4	23.28	0.995
PMMA	11520	112.3	26.28	0.999
HDPE	11507	44.27	22.68	0.981
Copper	24000	51.44	25.26	0.995
Aluminium	57601	125.8	26.56	0.993

The first evaluations of  $c_p$  have been done by Matlab software. A method of curve fitting was subsequently developed and implemented into the new software (Table 1). The input data is the cooling curves, which are obtained by a contactless temperature device (Figure 1). Data is provided in the form of text. The very good fit of results obtained by both methods of evaluation is clearly seen.

As we said, the time constant  $\tau$  is the basis for the determining of the material  $c_p$ . Equation 2, which represents the master equation of the problem, is linearized by logarithmic function (Figure 3). When the relaxation time  $\tau$  has been determined, this value is used to determine  $c_p$ .

In the second step, we start to find a further unknown physical value  $c_p$ . Because the equation for  $\tau$  in the form  $\tau = \rho c_p L / 2h_t$  ( $\rho$  is the density,  $L$  is the sample thickness and  $h_t$  is the total heat transfer coefficient) has an infinite number of solutions, which means that only fitting procedures can be used. The basic prerequisite is the knowledge or at least an estimate of the total heat transfer coefficient  $h_t$  from the material to the surrounding environment. This can be estimated if the

dimensions of the individual groups of samples (metals, plastics, laminates, etc.) are nearly the same, which in our case means a sample surface of about  $100 \text{ mm}^2$  and a thickness of approximately 2 mm. In this step, we can set  $h_t$  (15-20) according to the above described experimental conditions. For the further unknown ( $c_p$ ), we set a qualified estimate according to the type of material – metals, plastics, laminates, etc. (known from tables). The algorithm in the initial step is based on the intersection of the given and possible intervals of both quantities. Once the common intervals of meaningful values have been determined, the algorithm tries to find a single value using the interval division method by gradually decreasing the interval of both required values.

Every sample has been measured ten times; average values and 99 percent confidence intervals ( $P_{99}$ ) were calculated.

As we can see from the presentation of results, also the conformity of the measured or tabular data is very good (relative deviations are on the level of percent), both for low-conductive material, as well as for materials with relatively higher thermal transport properties. The values of  $P_{99}$  interval are calculated for chosen measured values. The width of  $P_{99}$  confidence interval is smaller than 10 percent in all samples under investigation.

**Table 2.** Statistical parameters of chosen materials ( $P_{99}$ )

Material	Measured $c_p$ [ $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ ]	$P_{99}$ [ $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ ]
Zn	$410.2 \pm 5.88$	(391.08 – 429.32)
PMMA	$1514 \pm 19.04$	(1452.11 – 1575.89)

In the Table 3 we present results from measurements of  $c_p$  realised on materials with a relatively wide spectrum of this physical value. Results cover such materials as copper, silicon polyurethane.

**Table 3** Measured values of  $c_p$  for different materials

Material	Measured $c_p$ [ $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ ]	Table value $c_p$ [ $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ ] [16]
Cu	387	383
Si	705	703
Pu	1480	1475

## CONCLUSIONS

In comparison with known methods our method offers:

- $c_p$  determination from the cooling curve of the tested sample
- The method is fully contactless and demands on the sample preparation are small.
- The presented apparatus is also able to measure materials with a large scale of thermal parameters.

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## REFERENCES

- [1] Maries Idicula, Abderrahim Boudenne, L. Umadevi, Laurent Ibos, Yves Candau, Sabu Thomas, Thermophysical properties of natural fibre reinforced polyester composites, *Composites Science and Technology*, Volume 66, 15, 1 (2006), 2719-2725.
- [2] Boudenne, Abderrahim, Ibos, Laurent, Boudenne, Abderrahim, Fois, Magali, Gehin, Evelyne, Majeste, Jean-Charles. Thermophysical properties of polypropylene/aluminum composites. *Journal of Polymer Science Part B: Polymer Physics*. Vol. 42, 4. (2004)722/732.
- [3] Ruiying Luo, Tao Liu, Jinsong Li, Hongbo Zhang, Zhijun Chen, Guanglai Tian. Thermophysical properties of carbon/carbon composites and the physical mechanism of thermal expansion and thermal conductivity, *Carbon*, Volume 42, 14, (2004) 2887-2895.
- [4] Malinarič, S., Contribution to the extended dynamic plane source method, *International Journal of Thermophysics*, 25, 6, 2004, pp. 1913-1919. [5] Almogbel M., and Bejan A., Conduction trees with spacing at tips, *International Journal of Heat and Mass Transfer*, Vol. 42, 1999, pp. 3739-3756
- [5] Naziev, Ya., Dz., Gasanov, V. ,G.: Determination of the Effect of Variability of the Thermal Properties of Materials When Measuring Thermal Conductivity Using Steady-State Thermal Methods, *Measurement Techniques*, 47, 1, (2004), 73-77.
- [6] Mandelis, A.: Progress in Photothermal and Photoacoustic Science and Technology, Elsevier New York Vol. 1 (1991).
- [7] Parker, W.,J., Jenkins, R.,J, Butler, C.,P.,Abbott, G.,L.: Method of Determining Thermal Diffusivity, Heat Capacity and Thermal Conductivity, *Journal of Applied Physics* 32 (1961) 1679.
- [8] Akoshima, M., Baba, T.: Thermal diffusivity measurements of candidate reference materials by the laser flash method, *International Journal of Thermophysics*, ( 2005), 26, 1, (2005) 151–163and *Thermodynamics*, Cape Town, South Africa, Paper DJ1, June 2004.
- [9] Gaal, P. S., THERMITUS, M. A., STROE, D. E.: Thermal conductivity measurements using the flash method , *Journal of Thermal Analysis and Calorimetry*, 78, (2004) 185–189
- [10] Mina,S., J. Blumm. J, Lindemann, A.: A new laser flash system for measurement of the thermophysical properties, *Thermochimica Acta*,455, 1–2, 1 (2007) 46–49.
- [11] Ezzahri, Y., Dilhaire, S., Grauby, S., Rampnoux, J.M., Claeys, W., Zhang, Y., Zeng, G., Shakouri, A.: Study of thermomechanical properties of Si/SiGe superlattices using femtosecond transient thermoreflectance technique. *Applied Physics Letters*. 5,9 (2005).1-3.
- [12] Lienhard, J., H., Phlogiston Press, Cambridge 2004.
- [13] Kopal, I. Košťial, P. Jančíková, Z. Valíček, J. Harničárová, M.: An analytical framework for the study of solid body cooling, *Defect and Diffusion Forum* 370, (2017),1-18.
- [14] Liňkov, J.,N.: *Lectures in mathematical statistics*. Parts 1 and 2. American Mathematical Society, 2005. ISBN 0-8218-3732-X.
- [15] Košťial P, Ružiak I, Jančíková Z 2012 *Patent* NO 303269 CS.
- [16] Brož,J.,Roskovec,V.,Valouch,M.: Physical and mathematical tables (in Czech) SNTL Praha 1990.