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Validation of the Thermal Diffusivity from Modified Monotonic Heating Regime Procedure[1]

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Problems of thermal diffusivity measurements when using monotonic heating regime methodology are discussed. A modification to a procedure applied to the specimen investigated at convective heat transfer conditions has been analyzed. Experiments are performed using two fluids of different thermal properties. By comparing the obtained data, it is possible to correct the negative effects of the finite Biot number. Results of test measurements performed on a PMMA cylindrical specimen are consistent with reference data, which demonstrates the reliability of the proposed modification. The suggested revisions in the measurement methodology have also been verified in the course of numerical simulations. It is concluded that high quality data can be obtained applying elementary experimental instrumentation and simple data processing.

KEY WORDS: FEM thermal modeling; monotonic heating regime methods; PMMA; thermal conductivity; thermal diffusivity.

1. INTRODUCTION

Regarding rather sophisticated and usually expensive apparatus needed for thermal diffusivity (TD) studies, the monotonic heating regime method [1] is one of the least expensive and operationally simplest ways of acquiring data for this essential thermal transport parameter. The method, especially in the case of a regular regime operation [1], presents many advantages. Most important is its versatility in terms of the specimen size, shape, and

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material type of low and medium thermal conductivity. However, the accuracy of the result is strongly dependent on the quality of replication of the assumed boundary condition. For the case of the assumed step rise of the specimen's surface temperature as a thermal load (Heaviside type first-order boundary condition), the main problem is to correct the errors resulting from a nonzero surface heat resistance effect. When in real experiments the investigated specimen is rapidly immersed in a certain fluid, it signifies a finite Biot number value ($Bi < \infty$). Corrections for that effect need a precise evaluation of the convection type, fluid properties, flow parameters, temperature distribution, etc. These data are rather difficult to obtain.

The idea of a possible modification overcoming these difficulties emerged during investigations described in Ref. 2. Using two thermostatic fluids of different properties, it is possible to eliminate almost entirely the negative effect of a finite Biot number.

The present paper deals with the problem of experimental and numerical validation of the modified procedure. The effect of the fluid properties and fluid flow changes has been studied analytically. Test measurements have been performed using water and ethanol as thermostatic fluids on polymethylmethacrylate (PMMA) specimens. Finally, the performance of the applied method has been confirmed using finite element modeling.

2. MODIFIED PROCEDURE SUMMARY

2.1. Background of a Finite Biot Number Effect Correction

The modification concerns the thermal diffusivity measured in a narrow temperature range with a sample heated or cooled to some constant temperature different from the initial value as described in Ref. 1. In such a case the solutions of heat transfer problems for basic geometries: an infinite plate, a cylinder, or a sphere, are expressed as a rapidly converging series in time [3]. The convergence results in a simplification of solutions in the regular regime[1] where temperature changes follow an exponential law [1,3];

$$\Theta(\xi,\tau) = A\left(\frac{\xi}{d}\right)\exp\left(-\frac{\tau}{\tau_{\mathrm{c}}}\right), \quad \tau_{\mathrm{c}} = f\left(\frac{1}{\kappa}, \frac{1}{h}\right) \tag{1}$$

where ξ is a dimensionless coordinate, τ is the time, d is a characteristic dimension, τ_{c} is a characteristic time, κ is the thermal diffusivity of

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[1] Usually the regular regime is distinguished by the condition $Fo > 0.4$, where Fo is the Fourier number [1].

the investigated medium, and h is the convection heat transfer coefficient. The characteristic time τ_c is a nonlinear function of κ and h. However, the form of the expression for τ_c suggests that, to a first approximation, it is inversely proportional to the thermal diffusivity and to the heat transfer coefficient, which means that it is directly proportional to adequate thermal resistances. It is true that the response time of the discussed system increases with a decrease in the thermal diffusivity/thermal conductivity and a decrease in the heat transfer coefficient of external convection.

Knowing τ_c and h, one can obtain the thermal diffusivity from formulae provided in Ref. 1. In practice, it is not so easy to determine a precise value of the heat transfer coefficient. The convection heat transfer effects can be neglected when $Bi > 100$ [1]. In other cases the finite Biot number effects should be corrected, for example, by applying multipoint temperature measurement data. The difficulties in evaluation of the finite Biot number effects result in reduced precision of the measurements.

To overcome these difficulties, a modification in the procedure has been proposed [4]. It is based on an assumption that, to a first approximation, the characteristic time can be described as

$$\tau_c = p\,\frac{1}{\kappa} + q\,\frac{1}{h} = \tau_{\mathrm{diff}} + \tau_{\mathrm{conv}} \tag{2}$$

where p and q are constants independent of the system thermal properties. The linear separation of conductive (τ_{diff}) and convective (τ_{conv}) heat transfer resistance terms seems to be valid when one of these is distinctly larger than the other. Now, performing experiments in two different convection circumstances and not knowing the precise values of heat transfer coefficients, but only the ratio,

$$r = \frac{\tau_{\mathrm{conv},1}}{\tau_{\mathrm{conv},2}} = \frac{h_2}{h_1} \tag{3}$$

the pure conduction term τ_{diff} can be described as [5]

$$\tau_{\mathrm{diff}} = \frac{r\,\tau_{c2} - \tau_{c1}}{r-1} = \tau_{c2} - \frac{\tau_{c1} - \tau_{c2}}{r-1} \tag{4}$$

where τ_{c1} and τ_{c2} are characteristic times recorded at two different experiments. The thermal diffusivity values are obtained from τ_{diff} and the appropriate formulae provided in Ref. 1.

The main advantage of the proposed modification is that the ratio r can be obtained much more easily than separate values of heat transfer coefficients h_1 and h_2. The precision is absolutely satisfactory assuming that the properties of the fluids are well known. And last but not least, parameters of the flow become insignificant if circumstances of the experiments are the same.

2.2. Practical Implementation of the Modified Procedure

Analyzing Eq. (4), one can realize that there are at least two requirements for a well-designed setup of an experiment:

(a) it is good to have a dominant conductance

$$\tau_{\text{diff}} > \tau_{\text{conv}} \text{ (or better } \tau_{\text{diff}} >> \tau_{\text{conv}}) \tag{5}$$

(b) the ratio r has to be distinctively different from unity

$$r \neq 1 \text{ (or better } r << 1 \vee r >> 1) \tag{6}$$

The first condition creates limitations for the thermal property, shape, and dimensions of the investigated specimen. However, the limitations are not so strict as in the classical procedure. The second condition can be easily fulfilled if similar baths of two fluids having distinctly different properties are used. In our case, water and ethanol have been used [5]. The differences of thermophysical properties of water and ethanol [6,7] result in differences in heat transfer coefficients of proportions 3.1 ($r \approx 3$) in the case when flow conditions are preserved similar. It is crucial that r has low sensitivity to temperature changes.

The effect could be illustrated with an example of calculations performed for a cylinder treated as a spheroid in external forced convection flow. A universal Yovanovich's formula for such a body can be applied to obtain the overall Nusselt number [8],

$$\overline{Nu_\delta} = \overline{Nu}_{\delta 0} + \left[0.15\left(\frac{l}{\delta}\right)^{1/2} Re_\delta^{1/2} + 0.35\, Re_\delta^{0.566}\right] Pr^{1/3} \tag{7}$$

where the characteristic dimension is defined as the square root of the spheroid surface area F,

$$\delta = \sqrt{F} = \sqrt{\pi\, d\, l + \pi\, \frac{d^2}{2}} \tag{8}$$

and $Nu_{\delta 0}$ is the overall Nusselt number in a no-flow condition (at zero flow speed), for a cylinder $\overline{Nu}_{\delta 0} = 3.444$ [8], Re is the Reynolds number related to δ , Pr is the Prandtl number, and l is the cylinder height (in the considered case). Next, one can calculate the values of the ratio r listed in Table I. As can be seen, the r parameter in this model is weakly dependent on the fluid

Table I. Results of Overall Nusselt Number Calculations (Eq. 5) and Ratios of the Appropriate Heat Transfer Coefficient Values for a Cylindrical Spheroid: Water vs. Ethanol[a]

Temperature(°C)	$\overline{Nu}_{\text{water}}$	$\overline{Nu}_{\text{ethanol}}$	$r = \frac{h_{\text{water}}}{h_{\text{ethanol}}}$
	$u_\infty = 5\,\text{cm} \cdot \text{s}^{-1}$		
10	81.97	86.07	2.9793
20	85.40	89.81	3.1294
30	88.46	94.88	3.2010
	$u_\infty = 20\,\text{cm} \cdot \text{s}^{-1}$		
10	172.22	180.96	2.9771
20	179.63	189.04	3.1273
30	186.25	199.96	3.1978

$\delta = 63.475\,\text{mm}$, $d = 15\,\text{mm}$, $l = 79\,\text{mm}$.

[a] Thermophysical data from Refs. 6 (water) and 7 (ethanol).

temperature and almost independent of the flow speed. Moreover, it maintains its value at a level of 3, which is another advantage of using water and ethanol as immersing media in view of the condition (6).

The most convenient way of performing measurements is switching the investigated specimen, with a temperature sensor mounted inside, between two strongly stirred baths of different temperatures: a water bath and an ethanol bath. From the recorded temperature histories, it is easy to derive the required characteristic times $\tau_{c1} = \tau_{c\,\text{ethanol}}$ and $\tau_{c2} = \tau_{c\,\text{water1}}$. After some correction [see Eqs. (3) and (4), Table I], the measured thermal diffusivity can be obtained based on the calculated τ_{diff} value [1].

3. EXPERIMENTAL

3.1. Experimental Apparatus

Many arrangements of the experiment are possible, even using one thermostatic bath, but including two thermostats into the measuring system and switching the investigated specimen/specimens between them seems to be the most convenient. The only inconvenience of this setup is the difference between the final temperatures of the two baths. This inconvenience could be overcome by repeating the experiments with exchanged temperatures of the two baths. There is no such problem when narrow temperature range investigations are considered.

The experiments discussed here were performed applying the two-thermostat apparatus with a simple data acquisition unit. A schematic diagram of the experimental installation is shown in Fig. 1. The thermo-

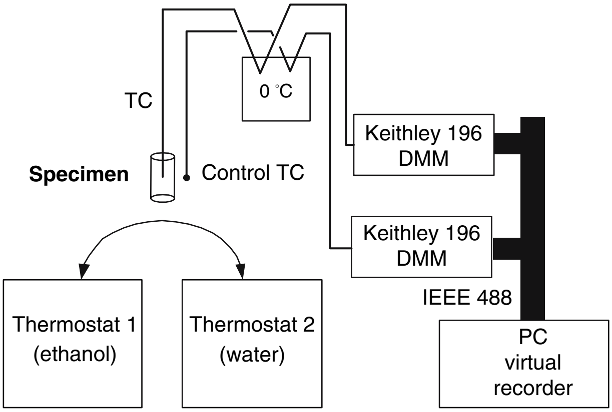

Fig. 1. Schematic diagram of the experimental apparatus.

stats, Lauda RL 6CP and ThermoHaake K35/DC50, are equipped with cooling systems enabling operation at subambient temperatures. Both of them are also provided with RS 232 interfaces. The two temperature signals—the first from the thermocouple measuring the temperature from inside the specimen and the second from the reference thermocouple (control TC) measuring the switched bath temperature—were referenced to the ice-water 0°C bath. Each thermocouple was connected to a digital multimeter (DMM) Keithley 196 model through the extension wires. The DMMs were controlled from the PC platform applying a National Instruments AT-GPIB IEEE 488.2 board and a virtual multichannel recorder. The recorder had been designed using a LabWindows software package. The temperature signal recordings were carried out with a maximum of $6^1/_2$ digit resolution.

3.2. Specimens and Measurement Procedure

The investigated material was polymethyl methacrylate (PMMA) Metapleks supplied by Dwory Oświęcim. The PMMA was selected for the test measurements because it is available, easy in treatment, and because its thermophysical properties are reproducible and relatively stable. The investigation results were analyzed against PMMA data provided by Salmon and Tye [9]. In order to make the comparison possible, some preliminary investigations of the Metapleks PMMA density and specific heat had been performed. The density was measured applying a Mettler Toledo AT 261 microbalance equipped with a density measuring kit. The specific heat was obtained in the course of microcalorimetric investigations

Table II. Results of Preliminary Investigations of Thermophysical Properties of the Metapleks PMMA

Property	Temperature	
Density (ρ) (kg·m^{-3})	19.5°C	1193.1
Specific heat (c_p) (J·kg^{-1}·K^{-1})	0–30°C	$1279 + 3.825T/°\mathrm{C}$

performed on a 20 mg sample. Measurements were carried out applying a Perkin–Elmer Pyris 1 differential scanning calorimeter (DSC) operating in a step heating/cooling mode [10]. The results are listed in Table II.

The main measurements were performed on cylindrical specimens with diameters of 15, 20, and 30 mm. The length/height of each specimen was about 80 mm. The specimens were cut out of a 100 mm in diameter cylindrical block of Metapleks PMMA. Each specimen was provided with a thermocouple placed in an axially drilled hole at the cylinder center (Fig. 2a). Tiny teflon tubing type K (from the Omega Company) thermocouples of about 0.05 mm diameter wires were applied for this purpose. The thermocouples were guided through ceramic tubes 1 mm in diameter. The void was filled with an epoxy to fasten and isolate the thermocouple end from fluid penetration. The thermocouples with lengths of about 0.5 m were extended with standard extension wires.

In the course of measurements the investigated specimen was moved at least six times from one bath into the other allowing a certain time for temperature stabilization. The water bath was set at 25°C and the ethanol bath at 15°C. Typical recorded signals from measurements of the 15 mm PMMA specimen are depicted in Fig. 3. The stabilization time was determined experimentally prior to the main measurements–the 15 mm PMMA specimen was immersed for at least 10 min, the 20 mm specimen for 20 min, and the 30 mm specimen for at least 30 min.

3.3. Experimental Data Processing

For further data processing the recorded signal (Fig. 3) was divided into subsequent heating or cooling segments as shown in Fig. 4a. Next, the time axis of each segment was scaled to zero at the beginning of the temperature changes in order to facilitate a subsequent approximation procedure. The approximation was applied only to the final part of the specimen's TC signal corresponding to the regular heat transfer regime. The beginning part of the temperature history, equivalent to 70% of the temperature rise or drop, was disregarded as shown in Fig. 4b. The same conditions of a regular heating regime (see e.g., Refs. 1 and 11) were

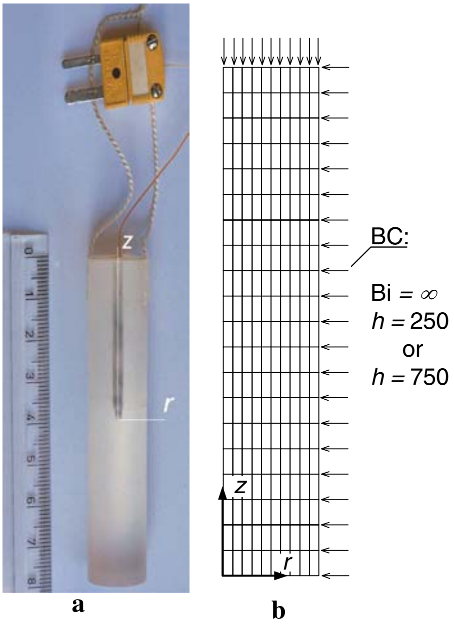

Fig. 2. Illustration of the investigated PMMA specimen: (a) the cylindrical specimen with a double Teflon tubing type K thermocouple and (b) finite element geometrical model of the specimen (the appropriate cylindrical co-ordinates are indicated in both pictures).

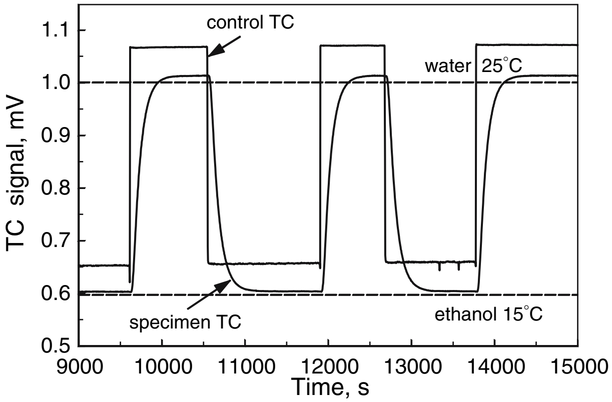

Fig. 3. Typical recording from 15 mm PMMA specimen measurements between 15°C ethanol and 25°C water baths.

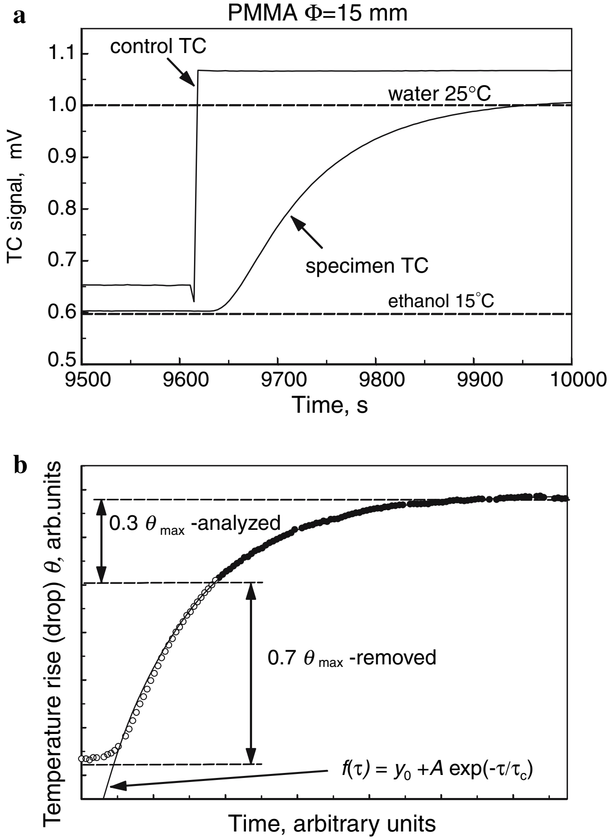

Fig. 4. (a) Example of a heating part of the signal selected for further data processing and (b) illustration of the characteristic time τ_c calculation procedure.

considered more than satisfactory. The 70% limit, neglecting the initial signal data, was taken after preliminary optimization analysis. Compliance with conditions for a regular regime mode, the problem of thermal resolution, and some operational effects were taken into consideration. The analyzed part of the specimen's TC signal was fitted with an exponential decay function,

$$f(\tau) = y_0 + A_0 \exp\left(-\frac{\tau}{\tau_c}\right) \tag{9}$$

A nonlinear least-squares data curve fitting procedure based on the Levenberg–Marquardt algorithm was applied. From the three approximation parameters, i.e., the level y_0, the amplitude A_0, and the characteristic time τ_c, only the last one was essential for further calculations.

In subsequent steps of data processing, the mean values of the corresponding parameters $\tau_{c1} = \tau_{c\ \mathrm{ethanol}}$ and $\tau_{c2} = \tau_{c\ \mathrm{water}}$ were calculated and the corrected characteristic time τ_{diff} was derived from Eq. (4) for each specimen. The ratio r for ethanol at 15°C and water at 25°C was evaluated to be $r = 3.29$. The thermal diffusivity values were obtained exploiting a standard formula for a finite cylinder from Ref. 1 expressed in the following form:

$$\kappa_{\mathrm{corr}} = \frac{1}{\tau_{\mathrm{diff}}}\left(\frac{\pi^2}{l^2} + \frac{23.132}{d^2}\right)^{-1} \tag{10}$$

where l is the specimen length, d is the diameter, and κ_{corr} is the measured thermal diffusivity corrected for finite Biot number effects.

3.4. Experimental Results and Discussion

The values of the thermal diffusivity obtained from Eq. (8) were referenced to 20°C, which was the mean temperature of the experiments. The results of calculations are listed in Table III, which contains the appropriate uncorrected values of the thermal diffusivity, calculated from the "water" and "ethanol" characteristic times τ_{c2} and τ_{c1}. In order to make the analysis easier, the results were converted to the thermal conductivity using the results from Table II and compared with the reference data provided by Salmon and Tye [9].

Commenting on the obtained results, it should be emphasized that relative differences from the reference data are not greater than 1.3% regarding the basic reference parameter, which is the thermal conductivity. Slightly greater differences in the thermal diffusivity are due to differences in the values of other parameters, i.e., the density and the specific heat (Table II, Ref. 9), applied in auxiliary calculations. Agreement of the obtained data with the PMMA reference results is illustrated in Fig. 5. The figure also shows the results of the "two fluid correction" procedure. The corresponding Biot numbers ranged from about 170 for the 30 mm specimen to about 290 for the 15 mm specimen in water experiments and from about 55 to about 90 in the analogous ethanol experiments. The diagram from Fig. 5 confirms these approximate calculation results.

In view of the sensitivity of the experimental thermal diffusivity to dimension changes (e.g., Eq. (8)), it is worth emphasizing that the results of measurements for the three different specimens are in agreement. The three measured specimens differed in diameters in the proportions 3:4:6.

Table III. Measured and Calculated Thermal Diffusivity and Thermal Conductivity in Comparisons with PMMA Reference Data [9]

	Thermal diffusivity, $\kappa \times 10^7 (m^2 \cdot s^{-1})$		
Tye and Salmon [9]	1.195		
Specimen diameter (mm)	Corrected	Water	Ethanol
30	1.177	1.145	1.082
20	1.164	1.143	1.101
15	1.182	1.163	1.123
	Thermal conductivity, $\lambda (W \cdot m^{-1} \cdot K^{-1})$		
	0.1904		
Tye and Salmon [9]	Corrected	Water	Ethanol
30	0.1900	0.1849	0.1747
20	0.1879	0.1846	0.1778
15	0.1909	0.1877	0.1813

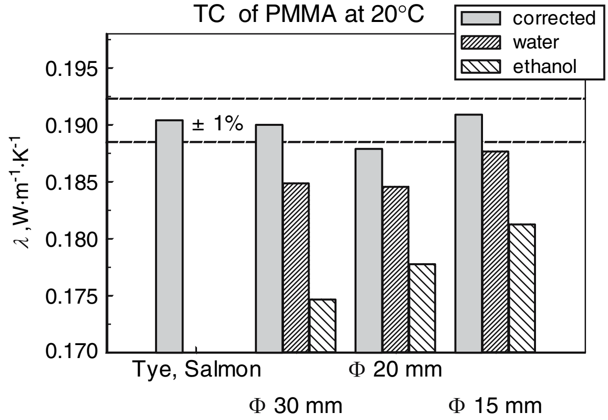

Fig. 5. Results of the Metapleks PMMA measurements converted to thermal conductivity in comparison with the reference PMMA thermal conductivity from Ref. 9 and illustration of effects of the correction procedure.

This means that the measured characteristic times were in proportion 9:16:36.

Regarding the whole discussion, the obtained results seem to be reliable.

4. NUMERICAL MODELING

4.1. FEM Modeling Procedure

Independent of the promising results of the experimental tests, additional numerical calculations were conducted in order to verify the proposed thermal diffusivity measurement procedure. A Cosmos/M finite element method (FEM) software package was utilized for this purpose. The performance of the package in reconstructing the modeled thermophysical parameters had been examined previously [12]. Because the present analysis is of a comparative nature, the numerical parameters of the model were optimized mostly to decrease the computation time. The numerical parameters and characteristics included the element type, the mesh density, the total modeled process time, the time step, and the solver type. The accuracy of optimized calculations in reconstructing the assumed thermophysical model properties was established to be about 3% for a Heaviside type first-order boundary condition (BC) load [12]. This number seems to be also valid for the monotonic type thermal loads.

For development of the model, an axial symmetry of the investigated system was assumed. The geometry of the thinnest 15 mm PMMA specimen was modeled applying two-dimensional (2D) planar four-node finite elements as shown in Fig. 2b. There were 20 elements in the axial direction and 10 elements in the radial direction in the mesh. The 15 mm specimen was selected since it represents the biggest challenge—for this specimen the contribution of convection in the modeled heat transfer phenomena (see Eqs. (2) and (5)) is the largest. To define the governing equation, pure conduction with no internal heat sources inside the specimen was assumed. The thermal properties of PMMA and their dependence on temperature were taken from Salmon and Tye [9].

The computations were carried out for three variants of boundary conditions of two types: first-order type, with a prescribed surface temperature and third-order, corresponding to convection on the surface with a prescribed fluid temperature and heat transfer coefficient. The details of the considered variants are as follows:

- the first variant, modeling a perfect, $Bi = \infty$ experiment with assumed surface temperature changes described by a Heaviside function (step function) with a temperature jump equal to 5°C and a final equilibrium temperature equal to 20°C;
- the second variant, modeling the ethanol immersion experiment, with an initial sample temperature of 15°C, a fluid temperature of 20°C, and a convective heat transfer coefficient equal to $250\,\mathrm{W{\cdot}m^{-2}{\cdot}K^{-1}}$;

- the third variant, modeling the water immersion experiment, with an initial sample temperature of 15°C, a fluid temperature of 20°C, and a convective heat transfer coefficient equal to 750 $W{\cdot}m^{-2}{\cdot}K^{-1}$.

The heat transfer coefficients were assumed to represent average experiment conditions according to the Nusselt numbers shown in Table I.

In the numerical modeling only heating experiments were taken into account. This was done in order to simplify the analysis. It should be mentioned that parallel computations for cooling from 25 to 20°C experiments were also conducted. The obtained results were consistent with the heating results.

4.2. Numerical Simulation Results and Discussion

The above stated problems were solved numerically utilizing a Cosmos/M FFT transient solver. The time step was set to 2 s, and the computations were conducted for 900 time steps. Further analysis was limited to temperature histories from the center of the specimen only. The simulated temperature signals are shown in Fig. 6.

Next, the numerical signals were processed in the same way as the experimental ones, which resulted in obtaining the appropriate characteristic times: the time constant of the perfect experiment τ_{Heav}, and the time constants of the ethanol immersion experiment τ_{c1} and the correspond-

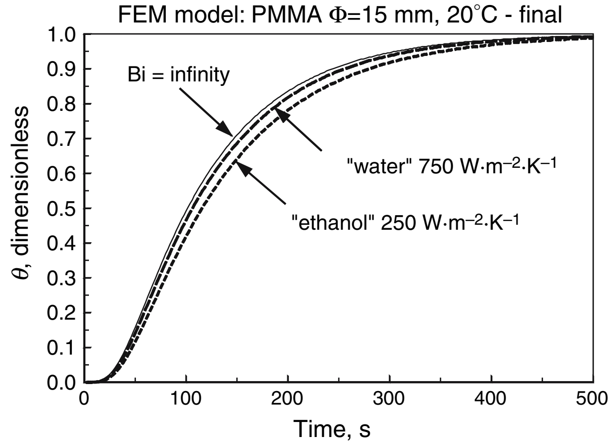

Fig. 6. Results of the numerical modeling - comparison between temperature histories from the three simulated experiments on the 15 mm PMMA specimen.

Table IV. Results of FEM Numerical Modeling—Comparison Between Characteristic Times of Temperature Equilibration

τ_{Heav}(s)	τ_{c1}(s)	τ_{c2}(s)	τ_{diff}(s)	$\frac{\tau_{\text{diff}}-\tau_{\text{Heav}}}{\tau_{\text{Heav}}}$(%)
87.429	100.556	91.663	87.781	0.40

Table V. Comparison Between the Assumed (Initial) Value of the Thermal Conductivity and Values Calculated from Numerical Models of the Perfect ($\text{Bi} = \infty$) and Real Modified Experiments

λ_{initial} $(\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$	λ_{Heav} $(\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$	$\lambda_{\text{modified meth.}}$ $(\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$
0.1904	0.18325	0.18266

ing water experiment τ_{c2}. On the basis of the derived numbers, shown in Table IV, the thermal diffusivity of the modeled PMMA specimen was calculated using the standard procedure for the Heaviside model [1] and applying a modified procedure for the next result. The results, converted to the thermal conductivity, are compared with the assumed specimen's thermal conductivity value (indicated as "initial") in Table V.

The numerical simulation revealed 4.2% underestimation of the transient heat conduction thermal properties obtained from the modified procedure when compared to the assumed value. But the results from the ideal experiment model are underestimated at nearly the same level of 3.9 %, which has been expected (see Section 4.1 and Ref. 12). The total difference can be attributed almost entirely to the numerical procedure. From the point of view of the conducted analysis, comparisons between the two analysed experiments are representative. For both cases: the thermal conductivity (Table V) and the corrected time constant (Table IV), the relative difference is equal to about 0.4%. The outcome is satisfactory which demonstrates the reliability of the proposed procedure for the analyzed experimental geometry and thermal load types.

5. CONCLUSIONS

A modified procedure of the monotonic heating regime measurement of the thermal diffusivity has been tested experimentally and analyzed in the course of numerical simulations. The modification is based on application of different fluids in the thermal load baths, which makes correction of the finite Biot number value effects possible (correction of convective heat transfer effects). Results of the performed tests are satisfactory. It has been demonstrated that a relatively high accuracy of the thermal diffusivity can be achieved using simple instrumentation and applying simple data processing. Comparisons of the measured polymethylmethacrylate thermal diffusivity with reference data [9] show differences of about 2.5%, while the differences for the thermal conductivity are about 1.5%. The results are promising, and the project is being extended to provide more data from quantitative analyses of the observed physical phenomena and to widen the range of application of the proposed method.

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