MEASUREMENT OF THERMAL DIFFUSIVITY UNDER CONDITIONS OF ORDERED HEAT TRANSFER.

Objective of the Exercise

A. Present the physical interpretation of thermal diffusivity as a coefficient of temperature equalisation in heat conduction phenomena.
B. To familiarise with the possibility of using special heat exchange conditions - in this case, phenomena associated with the so-called ordered heat exchange - to determine the thermophysical properties of substances.
C. To illustrate the relationship between thermal diffusivity and the time constants of thermal signals, as well as its connections with other thermophysical properties of substances, particularly thermal conductivity.
D. To present an example of compensating adverse metrological phenomena of complex heat exchange in the study of thermophysical properties.

1. Thermal diffusivity

Thermal diffusivity, also known as the temperature equalisation coefficient [5, 8, 9], or directly referring to the English term "thermal diffusivity" [4], is one of the fundamental thermophysical parameters. The basis for defining thermal diffusivity is the differential equation of unsteady heat conduction for an isotropic body. This is the previously cited Fourier-Kirchhoff equation, which in a Cartesian coordinate system takes the form:

$$\frac{\partial T}{\partial \tau} = a\nabla^2 T + \frac{1}{\rho c_p}\frac{\partial \lambda}{\partial T}\left[\left(\frac{\partial T}{\partial x}\right)^2 + \left(\frac{\partial T}{\partial y}\right)^2 + \left(\frac{\partial T}{\partial z}\right)^2\right] + \frac{q_V}{\rho c_p} \tag{1}$$

where: ρ is the density, c_p is the specific heat at constant pressure, T is the temperature, τ is the time, V is the volume, λ is the thermal conductivity, q_V is the volumetric efficiency of the internal heat source, x, y, z are spatial coordinates. Thermal diffusivity a is defined as

$$a \equiv \frac{\lambda}{\rho c_p} \tag{2}$$

From the above, it is evident that even when limited to the case of an isotropic medium, the direct physical interpretation of thermal diffusivity is limited to the proportional parameters presented in the defining relationship (2). Assuming thermal conductivity is independent of temperature:

$$\frac{\partial \lambda}{\partial T} = 0 \tag{3}$$

and in the absence of internal heat sources:

$$q_V = 0 \tag{4}$$

equation (1) takes the form of the Fourier differential equation [2, 10]:

$$\frac{\partial T}{\partial \tau} = a\nabla^2 T \tag{5}$$

The Fourier equation gives thermal diffusivity the physical sense of a parameter characterising the kinetics of heat exchange processes through conduction, simply the substance's ability to equalise temperature in heat conduction processes. This direct interpretation applies only to the case limited by conditions (3) and (4).

The concept of thermal diffusivity can also be introduced for orthotropic bodies where heat conduction is characterised by the thermal conductivity tensor λ_{ij} with three components. Generally, this imposes the constraint:

$$\frac{\partial \lambda_{ij}}{\partial T} = 0 \tag{6}$$

Complementarily with condition (3), to ultimately obtain the relationship:

$$\frac{\partial T}{\partial \tau} = a_{ij} \nabla^2 T \tag{7}$$

where:

$$a_{ij} \equiv \frac{\lambda_{ij}}{\rho c_p} \tag{8}$$

In the case of orthotropic bodies, obtaining analytical solutions to the heat conduction problem is much more difficult than for an isotropic medium. On the other hand, only an analytical solution can form the basis for developing a general method for studying thermal diffusivity.

2. Experimental Studies of Thermal Diffusivity

Fourier's equation (5) plays a crucial role in the direct determination of the thermal diffusivity. Most experimental methods of this type use solutions to problems described by Equation (5) along with additional formulated boundary conditions (both boundary and initial) [2-4, 6, 8]. Methodological differences are essentially limited to the type of boundary conditions assumed in the model, possible simplifications of the obtained solutions, and the way the model assumptions are implemented in a real experiment. It is worth noting that in terms of the geometry of the model, Fourier's equation (5) is general in nature: it can be applied not only in the Cartesian coordinate system, but also in cylindrical and spherical systems, depending on the form of the Laplacian $\nabla^2 = \Delta$ (see [10], p. 27).

Indirect methods provide an alternative to direct methods. In this case, experimental studies determine density, specific heat at constant pressure, and thermal conductivity. The thermal diffusivity is calculated using Equation (2). In the extended interpretation of thermal diffusivity, the variability of individual thermal properties with temperature is considered:

$$a(T) = \frac{\lambda(T)}{\rho(T) c_p(T)} \Leftrightarrow \lambda(T) = \rho(T) \cdot a(T) \cdot c_p(T) \tag{9}$$

The question of the variability of thermophysical parameters with temperature and the impact of this variability on the results of thermal characteristics according to equation (9) falls within the broader class of issues related to the thermal resolution of measurements of thermal properties measurements [5]. A detailed discussion of these issues is beyond the scope of this study. A brief comment should simply state that, generally, specific heat, thermal conductivity, and thermal diffusivity exhibit the greatest relative variability. Changes in these properties should be considered when temperature changes exceed a few tens of kelvins at room temperature. Changes in density become significant with temperature differences on the order of several hundred kelvins.

Since the method of studying thermal diffusivity under conditions of orderly heat exchange, also known as the method of monotonic thermal excitation (see [11]), is a direct method, attention will be paid to it in the following sections. In addition to characterising both methods, it should be noted that the main disadvantage of indirect methods is the propagation of measurement errors of each component quantity, affecting the accuracy of the calculated diffusivity. Furthermore, it is important to ensure the correlation of methods to determine density, specific heat, and thermal conductivity so that the diffusivity calculated based on expression (9) retains its character as an instantaneous property. This primarily concerns maintaining the appropriate thermal (temperature) resolution of the measurement (see, e.g., [5]).

Direct methods are mostly free of the above-mentioned disadvantages. Unlike time-consuming thermal conductivity measurements, experiments do not take too long. In most cases, measurements can be performed on very small samples, with dimensions on the order of millimetres, and high resolution can be achieved. However, determining thermal diffusivity based on recorded signals requires the use of rather complex data processing procedures, both mathematically and numerically. Since it is necessary to study time-varying signals, the research systems are relatively expensive and complex.

Applying the direct method of measuring thermal diffusivity involves analysing the time-varying temperature field with time variations, which follows from the nature of equation (5). Thus, in addition to the problem of recording thermal signals, the issue of thermal excitation must be addressed. Depending on how thermal excitation is implemented, direct methods can be divided into [4]:

I. Methods of unsteady/transient nonperiodic states:

- Impulse methods;
- Monotonic methods;
- Transient state methods with other aperiodic excitation (hot wire, hot disk/hot disk, flat source, rectangular, etc.).

II. Methods of periodic excitations:

- Thermal wave methods;
- Periodic excitations using electron beams.

Group I includes impulse methods [3, 4, 9] and methods of monotonic thermal excitation [3, 11]. The second group includes thermal wave methods and methods where the excitation source is an amplitude-modulated electron beam. In the first case, the basis for distinction is the measurement methodology, while in the second group the differences are mainly in the construction of the apparatus and the associated different research scopes. The thermal wave method is mainly used in low- and medium-temperature studies, while the electron-beam bombardment method, similar to the impulse method, dominates high-temperature measurements.

The subject of the exercise is the measurement of the thermal diffusivity using a modified method of monotonic thermal excitation with step excitation (Heaviside). Monotonic thermal excitation methods, that is, methods for studying under conditions of orderly heat exchange [10, 11], are characterised by relatively simple experimental methodology and instrumentation for measurements carried out at low temperatures[1]. Unfortunately, this is associated with increased measurement errors and the limitation of the range of tested materials to those with relatively low thermal conductivity for medium temperature studies[2]. The relative error of typical measurements ranges from 2% to even 12%, with typical inaccuracies of 1.5% to 5% for measurements conducted under comparable conditions by the impulse method, 1% to 9% by the thermal wave method, and 2% to 10% by the electron bombardment method.

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[1] This does not apply to high-temperature studies or studies conducted in the cryogenic temperature range.

[2] Primarily, this pertains to research within the typical operational ambient temperature range, from a few dozen degrees Celsius below zero to several dozen or a couple of hundred (one hundred to two hundred) degrees above zero.

3. Ordered Heat Transfer in the Context of the Monotonic Thermal Excitation Method

The theoretical basis of monotonic methods is the theory of ordered heat transfer formulated by Kondratyev [10]. In the English literature, this theory is commonly referred to as the "regular heating regime" ([4] p. 304 / [11]).

Kondratyev's theory is based on the observation that initial conditions influence the development of the temperature field and temperature changes only in the initial stage of the temperature equalisation process. After the transient period, the temperature field and temperature changes depend solely on the properties of the object under study and the boundary conditions, which also include the properties of the surroundings. When temperature changes on the boundary of the considered object have a regular character, analysing the temperature changes within the object allows the determination of individual thermophysical parameters, including thermal diffusivity.

The mathematical justification for the theory of ordered heat transfer is based on the analysis of solutions to unsteady heat transfer problems. Using Fourier series theory, a typical analytical solution with separated variables for step excitation can be expressed as [2]:

$$\theta(\xi,\tau) = \sum_{n=1}^{\infty} A_n \Phi_n(\xi,\mu_n) \exp\left[-\mu_n^2 \frac{a\tau}{l^2}\right] = \sum_{n=1}^{\infty} A_n \Phi_n(\xi,\mu_n) \exp[-\mu_n^2 Fo] \tag{10}$$

where $\theta(\xi,\tau)$ is the dimensionless temperature (normalised temperature excess), which is a temperature relative to, for example, a given characteristic temperature or changes in maximum temperature:

$$\theta(\xi,\tau) = \frac{T(\xi,\mu_n) - T_{min}}{T_{maks} - T_{min}} \tag{11}$$

T_{max} i T_{min} are the maximum and minimum temperatures, respectively, ξ\) is a spatial variable associated with the distinguished direction, *l* is the characteristic dimension of the object in the given direction (e.g., thickness of a plate, radius of a cylinder, etc.), (A_n) is a sequence of constants, (Φ_n) is a sequence of functions dependent only on spatial variables, (μ_n) — is an increasing sequence of numbers:

$$\mu_1 < \mu_2 < \mu_3 < \cdots \tag{12}$$

which are solutions to the characteristic equation associated with the given heat transfer problem, while *Fo* is the Fourier number:

$$Fo = \frac{a\tau}{l^2} \tag{13}$$

A notable feature of the functional series (10), due to the strong monotonicity properties of the numerical series (12) and the presence of the series terms μ_n, in the exponents of the subsequent exponents raised to the power of 2, is the rapid decrease in the influence of higher-order components as time τ increases. For τ exceeding a certain specified value, the solution

$$\theta(\xi,\tau) = A_1\Phi_1(\xi,\mu_1)\exp\left[-\mu_1^2\frac{a\tau}{l^2}\right] + A_2\Phi_2(\xi,\mu_2)\exp\left[-\mu_2^2\frac{a\tau}{l^2}\right] + A_3\Phi_3(\xi,\mu_3)\exp\left[-\mu_3^2\frac{a\tau}{l^2}\right] + \cdots \tag{14}$$

can be approximated by a first-order approximation [10, 11]:

$$\theta(\xi,\tau) \cong A_1\Phi_1(\xi,\mu_1)\exp\left[-\mu_1^2\frac{a\tau}{l^2}\right] = A_1\Phi_1(\xi,\mu_1)\exp[-b\tau] \tag{15}$$

where *b* is the temperature change rate (heating/cooling rate):

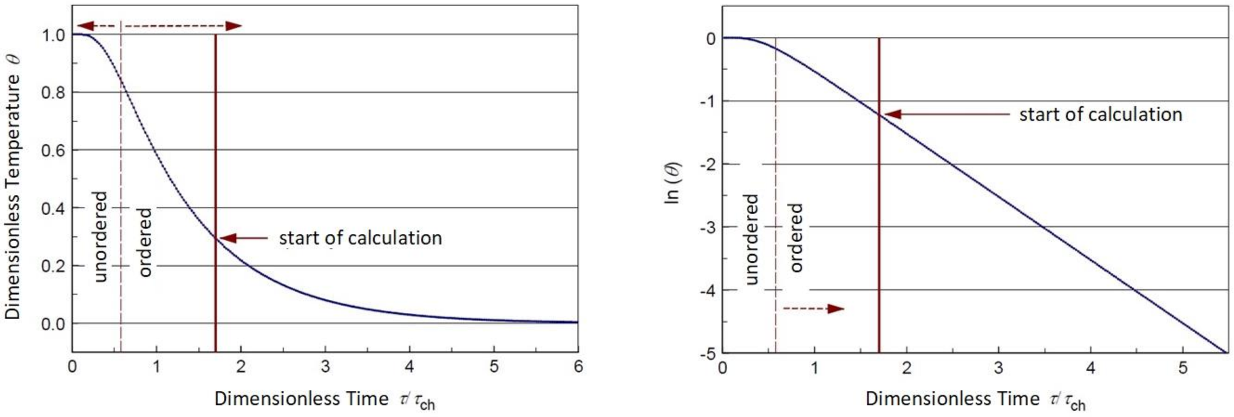
$$b = \mu_1^2\frac{a\tau}{l^2} = \frac{1}{\tau_{ch}} \Rightarrow a = \frac{l^2}{\mu_1^2}\tau_{ch} = \frac{l^2}{\mu_1^2}\frac{1}{b} \tag{16}$$

The rate of temperature change is the reciprocal of an arbitrarily introduced characteristic time of the temperature equalisation process τ_{ch}.

In ordered heat transfer ($\tau > \tau_u$) the course of normalised temperature excess (14) under step excitation therefore approaches the exponential course (15). The literature provides the following condition for the regular heat transfer regime [11]:

$$Fo > Fo_u = \frac{a \cdot \tau_u}{l^2} = 0{,}4 \tag{17}$$

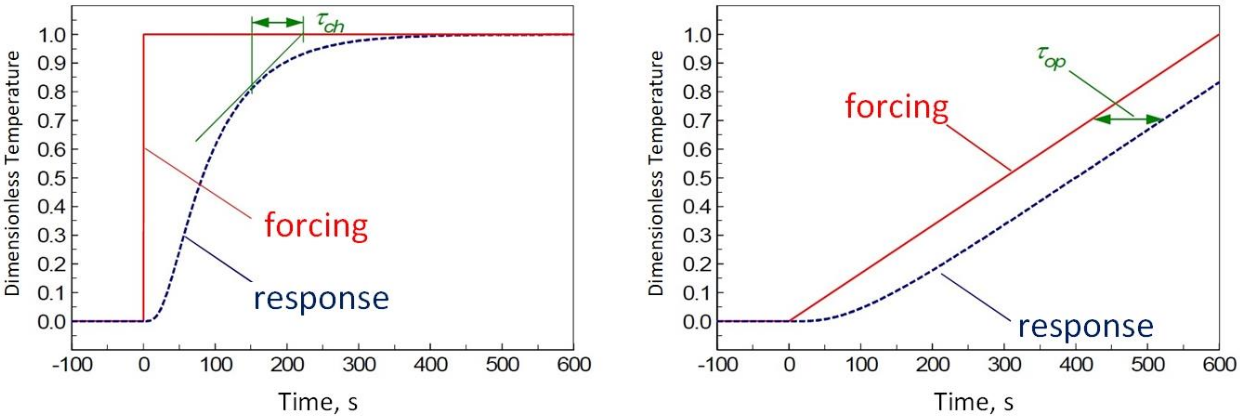
An example of changes over time in the normalised temperature excess is shown in Figure 1. The coefficient *b, the* rate of change rate—contains information about the properties of the body, reduced in the case described by Fourier's equation to thermal diffusivity and boundary conditions. Its determination does not pose significant difficulties. This can be done using an approximation of the course with a function containing an exponential term (Fig. 1.a) or by analysing the logarithmic signal. In the latter case, determining the coefficient *b* reduces to finding the slope of the linear segment that overlaps the measurement points in the ordered heat transfer area (Fig. 1.b; a method often used in the graphical analysis of measurement signals).



a

b

Fig. 1. Illustration of the normalised temperature excess for a cylinder subjected to a step surface temperature change: a, changes in temperature as a function of dimensionless time, and b, logarithmic plot of temperature changes.



a

b

Fig. 2. Comparison of (a) step and (b) linear excitation in monotonic heating/cooling methods.

4. Monotonic Thermal Excitation Method with Step Excitation and Modified "Two-Fluids" Method

Monotonic thermal excitation can be implemented in various ways. In typical cases, the above-mentioned step and linear excitations are used [11] (see Fig. 2). It is possible to implement other types of monotonic excitation, but only the two mathematical models mentioned at the beginning fall within the class of problems discussed in the previous chapter. Studies with step excitation (Heaviside) are characterised by a particularly simple methodology.

4.1. Classic-Step Excitation Method

For temperatures slightly deviating from room temperature, "step" measurements of diffusivity are performed by placing the stabilised and temperature-equalised test object into a fluid of a different temperature. The initial condition thus takes the form:

$$T(\boldsymbol{r},\tau)|_{\tau=0} = T(\boldsymbol{r},0) = T_0 \tag{18}$$

where **r**=[x, y, z] is the position vector. The boundary condition on the surface *S* is of the third kind (Newton's law [11]):

$$\left.\frac{\partial T}{\partial \boldsymbol{n}}\right|_S = -\frac{\alpha}{\lambda}\left(T|_S - T_p\right) \tag{19}$$

where ***n*** locally denotes the normal direction to the surface *S*, α is the heat transfer coefficient and *T* is the fluid temperature. The cooling rate in this case depends both on the thermal diffusivity *a* and the heat transfer coefficient α, expressed by the Fourier number (13) and the Biot number:

$$Bi = \frac{\alpha \cdot l}{\lambda} \tag{20}$$

During the experiment, the temperature change of a selected point in the body is recorded. After determining the rate of temperature change under ordered heat transfer *b* or, alternatively, the time constant τ_{ch}:

$$b = \frac{1}{\tau_{ch}} = f(Fo, Bi) \tag{21}$$

Thermal diffusivity can be calculated. Further analysis is based on solutions to initial-boundary problems with Fourier's equation with solutions in the general form (10) or (15) after simplification. Problems are formulated for the following model geometries [6, 11]:

I. A flat infinite plate of thickness 2δ.
II. An infinite cylinder with radius *R* and diameter *D* = 2*R*.
III. A sphere with radius *R* and diameter *D* = 2*R*.
IV. A cylinder with radius *R* and length 2*l*.
V. A rectangular prism with dimensions $2l_1$, $2l_2$, and $2l_3$.
VI. A cylinder with *R* and length *2l* made of orthotropic material with two different components of the diffusivity tensor and an axis orientated perpendicular to the plane defined by the main directions with equal diffusivity values.
VII. Three rectangular prisms of orthotropic material with edges aligned with the main axes of the material texture and edge proportions of 1:2:3, 1:2:2, and 1:1:2, respectively.

Solutions are sought for selected, characteristic points of the object. Typically, this is the central point defined by the intersection of the symmetry axes of the object analysed.

A. The problem solution is determined for a finite Biot number, and then the value of this number is calculated on the known properties of the fluid and the experimental conditions (convection heat transfer conditions on the surface of the analysed object). Introducing the calculated value into the obtained solution allows the number of unknowns in Equation (20) to be reduced to one: the Fourier number *Fo* containing thermal diffusivity.

B. An infinite Biot number is assumed, and the experimental conditions are adjusted accordingly. For an infinite Bi value, which in practice means satisfying the condition:

$$Bi > 100 \tag{22}$$

(see [11] - Meeting this condition guarantees 2% measurement accuracy of *a*), the third-kind boundary condition asymptotically transforms into a first-kind condition:

$$T|_S = T_p \tag{23}$$

Formulas to convert the rate of temperature change (or alternatively the characteristic time) to the material of the diffusivity of the tested object are provided in Table 1.

In practical research, determining the exact value of the Biot number using Method A is very difficult. Ensuring conditions that satisfy inequality (21) is also challenging, and even this does not completely eliminate errors associated with thermal resistance in heat transfer on the surface of the test sample.

Table 1.

Geometry of the Object	Formula
I. Flat plate	$a = \frac{4l^2}{\pi^2} b$ where: l is half the thickness of the plate
I II. Infinite cylinder	$a = \frac{4l^2}{\pi^2} b$ where: l is half the thickness of the plate
III. Sphere	$a = \frac{R^2}{5.783} b$ where: R is the radius of the cylinder
IV. Finite cylinder	$a = \frac{b}{\frac{5.783}{R^2} + \frac{\pi^2}{4l^2}}$ where: l is half the height of the cylinder with radius R
V. Rectangular prism	$a = \frac{b}{\frac{\pi^2}{4}\left(\frac{1}{l_1^2} + \frac{1}{l_2^2} + \frac{1}{l_3^2}\right)}$ where: l_1, l_2, l_3 are half the corresponding edge lengths
VI. Orthotropic rectangular prism	$a_1 = 6{,}76\, l^2 \,(27b_1 - 7b_2 - 5b_3) \cdot 10^{-3}$ $a_2 = 135{,}3\, l^2 \,(b_3 - b_2) \cdot 10^{-3}$ $a_3 = 730{,}2\, l^2 \,(b_2 - b_1) \cdot 10^{-3}$ where *2l* is the length of the shortest edge of the rectangular prisms with edge length proportions 1:2:3, 1:2:2, and 1:1:2, and b_1, b_2, b_3 are the rates of

	temperature change determined in experiments carried out for the mentioned rectangular samples.

4.2. Modification of the Method Using a Two-Fluids Bath

In the modified procedure, errors due to the finite Biot number are corrected [6]. This is achieved using the results of two experiments conducted under convective heat transfer conditions with different values of the heat transfer coefficient α, ensuring that the following defined proportionality coefficient is different from one:

$$1 \neq k = \frac{\alpha_1}{\alpha_2} \tag{24}$$

These could be identical experiments carried out using two fluids with different physical properties [6, 7, 8]. When determining the thermal diffusivity from the appropriate formulas (Table 1), coefficient *b* are replaced by the values calculated based on the corrected characteristic time of complementary experiments [5, 6]:

$$\tau_{kor} = \frac{k \cdot \tau_1 - \tau_2}{k - 1} \tag{25}$$

$$b = \frac{1}{\tau_{ch}} \tag{26}$$

Times τ_1 i τ_2 are the characteristic times of temperature equalisation after immersion in the bath of the first and second fluids, respectively. The correction procedure corresponds to introducing a correction for the finite Biot number, i.e., a correction accounting for thermal resistance in heat transfer phenomena. The values of the proportionality coefficient for the case where fluid 1 is water and fluid 2 is ethanol are provided in Table 2.

Table 2. Values of the proportionality coefficient $k = \frac{\alpha_{water}}{\alpha_{ethanol}}$ for identical forced convection velocities of water and ethanol as working fluids (data from [7])

Temperature [°C]	Proportionality coefficient *k*
0	2,85
10	2,98
20	3,13
30	3,19
40	3,26
50	3,30

5. Measurement System and Testing Procedure

The optimal method for measurements using the modified monotonic thermal excitation method involves alternating experiments by immersing the test sample in baths of two different fluids. To conduct diffusivity tests, it is essential to enable:

- placing the test sample in fluid environments with constant temperatures;

- measuring and recording the temperature values with appropriate accuracy and sampling frequency.

For this purpose, a measurement system was set up consisting of (Fig. 3):

- two thermostats (low temperature ThermoHaake DC50 K35, low-temperature Lauda RL6CP or thermostats UTU-2, UTU-4);

- a data acquisition and processing card for thermoelectric measurements (twelve-channel NI 4350 card with TC 2190 terminal and USB interface, six-channel NI 4350 card with PCIMC connection, or six-section Keithley DAS TC card);

- a personal computer.

The samples tested can typically be in the shape of a cylinder (see Fig. 4), sphere, flat plate, or rectangular prism. For nonstandard cases, measurements can be performed on samples with irregular shapes. Temperature measurement in the experiment is carried out using type K thermocouples. Virtual instruments can be used to control the operation of the measurement system. The measured temperature change curves are recorded in the computer's memory as text files.

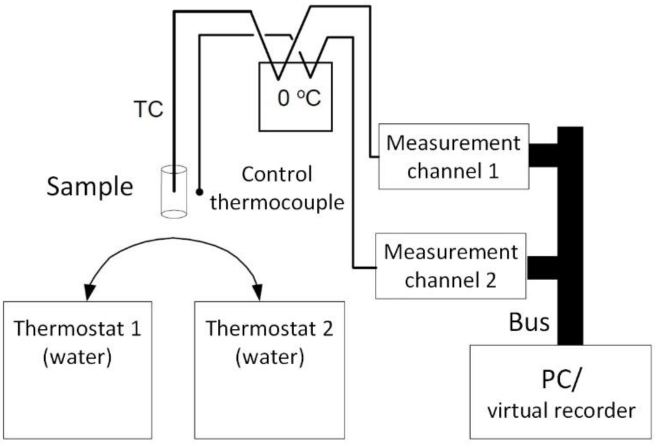


Fig. 3. Schematic of a sample measurement system for thermal diffusivity studies using the "two-fluids" method.

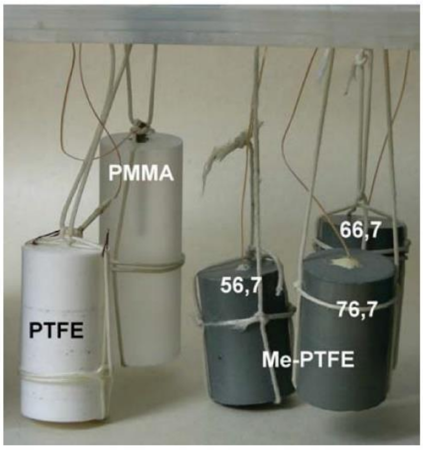


Fig. 4. View of samples suspended on a holder and prepared for immersion testing.

Fig. 5. View of thermostat baths (illustration of the testing procedure).

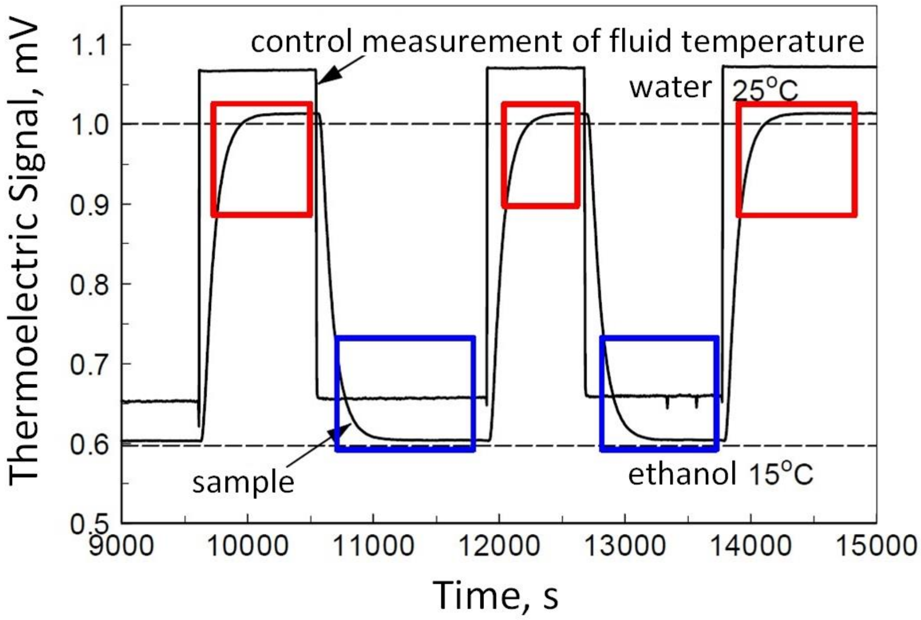


Fig. 6. Example of signal recording from immersion tests: the rectangles indicate the signal segments used to calculate the characteristic times of the temperature equalisation process after immersion in water (red) and ethanol (blue).

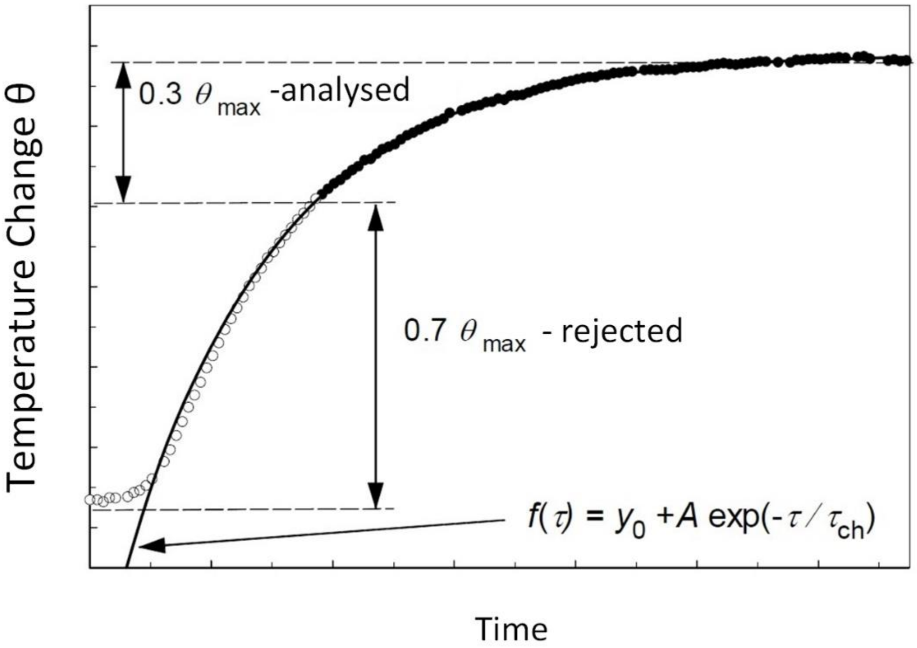


Fig. 7. Illustration of the data processing method for the temperature rise signal.

As indicated previously, during the tests, the sample (or samples) is placed in the bath of fluid 1. After the temperature stabilises, it is transferred to the bath of fluid 2 at a different temperature (Fig. 5). The stabilisation and transfer procedure of the sample can be repeated several times (Fig. 6); however, two immersions are sufficient to determine the two elementary characteristic times. For analysis, a segment of the signal corresponding to the final 30% of the relative temperature drop or rise is "cut out" (see Fig. 7). The data are approximated by the function:

$$f(\tau) = y_0 + A \cdot e^{\left(-\frac{\tau}{\tau_{ch}}\right)} \tag{27}$$

Any data processing software can be used for calculations. However, it should be noted that a crucial element of the numerically implemented procedure is the preliminary rescaling of time to start at zero (subtracting the value corresponding to the first entry of the time column from each entry). After determining the characteristic times, the measured results can be used to calculate the time constant τ and diffusivity from formula (24), followed by the temperature change rate (25) and the diffusivity of the sample based on the appropriate relationship from Table 1 according to its type (sample shape). For repeatable studies, average times are used for calculations according to these formulas (24).

6. Experimental Procedure

After the instructor provides the recommended instrument settings, you should:

Preparation for Testing

1. Prepare the test logs.

2. Measure the dimensions of the test sample(s).

System Setup

3. Turn on the thermostats and set the specified temperatures (recommended: water 20°C, ethanol 10°C).

4. Connect the ends of the measurement thermocouples (sample and control thermocouples) to the data logger block.

5. Turn on the computer and start the data acquisition programme.

Performing the measurement

6. Immerse the sample(s) in bath 1 and wait until the temperature of the sample stabilises (usually no longer than 20 minutes).

7. Transfer the sample to bath 2 and wait until its temperature stabilises.

8. Repeat the steps two to five times.

Repeating the Measurement in Modified Conditions

9. After changing the thermostat settings as agreed with the instructor, repeat the tests.

Data Processing

10. Determine the characteristic time value for each immersion experiment by analysing the signal segments corresponding to the final 30% of the temperature rise/fall.

11. Calculate the average characteristic times of immersion experiments for each material tested.

12. Determine the corrected time using the calculations from Step 9, times, relationship (24), and the average values of the proportionality coefficients from Table 2.

13. Calculate the rate of change in temperature using formula (25) and the diffusivity of the test sample material using the appropriate relationship from Table 1 corresponding to its type (sample shape).

The prepared report should include information on the conditions of the tests performed, covering recorded measurement signals, results of characteristic time calculations, temperature change rate, thermal diffusivity, and, if provided by the instructor, data on other thermophysical parameters of the tested material. The report should be supplemented with diagrams that illustrate the results of the diffusivity measurement and the results of the error analysis.

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