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A New Method of Simultaneous Measurement of Thermal Properties by Iterative Calculations*

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A new method of simultaneous measurement of thermal properties — thermal conductivity, thermal diffusivity and heat capacity — of plane-shaped solids by continuous heating is presented.

The principles of the measurement are based on the iterative calculations, that is, the numerically calculated temperature responses of a specimen with assumed thermal properties are compared with the experimental ones, and the above procedure is iterated until the calculated temperature responses coincide with the experimental ones.

Necessary conditions of measurement and calculation are determined by theoretical analysis.

This method is examined theoretically and experimentally and is shown to be useful for rapid estimation of temperature dependencies of the above thermal properties.

1. Introduction

The principle and procedure of measuring the properties by iterative calculations are illustrated by a flow chart in Fig.1 for the case of determination of thermal properties of material. First, the properties are assumed to have certain values, and the fundamental equation of heat conduction is solved numerically from the above assumed properties by a computer, where the initial and boundary conditions are determined from the temperature measurements of a specimen. The calculated temperature responses in the specimen are compared with the corresponding experimental temperature. The assumed values of the properties are reassumed to iterate, until the calculated and the experimental temperature responses agree with each other with sufficient accuracy, and when both responses coincide, the assumed properties at that value are considered the experimental results by iterative calculations.

The principle of most usual methods of measuring properties is based on simple analytical solutions and the apparatus for measurement have to be made carefully under mathematically defined boundary conditions. On the other hand,

in the method of the measurement by iterative calculations based on the numerical solutions, the apparatus for measurement can be composed of convenient configurations and the procedure of the measurement is simpler.

The principle in measuring procedure of thermal diffusivity by iterative calculations was investigated by Carter, R.L., et al.⁽¹⁾ or Beck, J.V.⁽²⁾ In the present paper, we would like to extend the iteration method to simultaneous determination of thermal properties — thermal conductivity, thermal diffusivity, and heat capacity.

Recently numerical methods⁽³⁾ using a computer made it possible to solve any nonlinear heat conduction problem in which the temperature dependencies of the thermal properties are considered, but the methods for measurement of the thermal properties which are essential to solve

Fig.1 Flow chart of the method of measurement by iterative calculations

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the above problems are unsatisfactory. In the usual methods of measurement as mentioned in Refs.(4) and (5), the transient methods of heating small specimens by step or pulse and heating plane-shaped specimens continuously are found suitable to obtain the temperature dependencies of the thermal properties in short measuring time. A method of pulse-wise heating by the laser, investigated by Takahashi, Y., et al.⁽⁶⁾ and one of step-wise heating by xenon lamp, investigated by Kobayashi, K., et al.⁽⁷⁾ are known to be useful for the measurements at high temperature. As continuous heating methods, a method of measurement of thermal conductivity by heating thin specimens continuously has been investigated by Eiermann, K., et al.,⁽⁸⁾ a method of measurement of thermal diffusivity by continuous heating⁽⁹⁾ and a method of simultaneous measurement of the thermal properties by continuous heating⁽¹⁰⁾ have been investigated by the authors.

Usual methods of measurement of the thermal properties are classified by the principle of measurement and the heating method, as shown in Table 1, and the classification of the methods of transient measurement is shown in Table 2.

In the present paper, method of measurement of the thermal properties by iterative calculations is extended to simultaneous measurement of thermal conductivity, thermal diffusivity and heat capacity; and the principle of measurement is based on the comparison methods of continuous heating improved from our previous method⁽⁹⁾ of thermal diffusivity by continuous heating. In Fig.2(a) a plane-shaped specimen is placed between two standard specimens of which

(a) Co-ordinate system

(b) Temperature rise in the specimen

Fig.2 Co-ordinate system and temperature rise in the specimen

Table 1 Classification of the methods of measuring thermal properties

Steady method	{ Absolute method ⁽¹¹⁾ Comparison method ⁽¹²⁾	
	{ Pulse-wise heating Step-wise heating Periodic heating Continuous heating	
Transient method	{ Methods based on analytical solutions Methods based on iterative calculations	

Table 2 Classification of the transient measuring methods

Principle of measurement	Configuration of heat sources		References	Obtainable thermal properties	Kind of specimen	Necessary thermal Properties of standard specimen
Method based on analytical solutions	Line source	Step-wise heating	Pfriem,H. ⁽¹³⁾	λ	Gas, Liquid Powder	
	Non-contact heating	Pulse-wise heating	Takahashi,Y. et al. ⁽⁶⁾	$\alpha \lambda cp$	Solid	$(cp)_s$
		Periodic heating	Wheeler,M.J. ⁽¹⁴⁾	α	Solid	
	Radiation Laser Electric beam	Step-wise heating	Kobayashi,K. ⁽⁷⁾	$\alpha \lambda cp$	Solid	$(cp)_s$
	Plane source	Step-wise heating	Krischer,O. ⁽¹⁵⁾	$\alpha \lambda cp$	Solid	
			Authors ⁽¹⁶⁾	$\alpha \lambda cp$	Solid	
			Authors ⁽¹⁷⁾	$\alpha \lambda cp$	Solid, Liquid Powder	$\sqrt{(\lambda cp)_s}$
	Plane source	Continuous heating	Eiermann,K. et al. ⁽⁸⁾	λ	Solid	$(cp)_s$
			Authors ⁽⁹⁾	α	Solid, Powder	
Authors ⁽¹⁰⁾			$\alpha \lambda cp$	Solid		
Method based on iterative calculations	Cylindrical source	Step-wise heating	Carter,R.L. et al. ⁽¹¹⁾	α	Solid, Powder	
	Plane source	Step-wise heating	Beck,J.V. ⁽²⁾	α	Solid, Powder	
		Continuous heating	The present paper	$\alpha \lambda cp$	Solid, Powder	$\alpha_s \lambda_s$

the thermal properties are assumed to have been determined accurately, and the test specimens are heated so that the temperatures on both the surfaces ($x=\pm L$) may be raised smoothly with time. The heat flux into the specimens is determined from the temperature change in the standard specimens and the iteration method is applied to the temperature responses of the test specimen by this heat flux. Such method of measurement has the following merits: ① the temperature dependencies of the thermal properties can be determined simultaneously in short measuring time; ② direct measurement of heat flux into the test specimen is not necessary; and ③ the heating of specimens is arbitrary. These merits are attributable to the way of the continuous heating, the way of comparison with the standard specimen, and the way of the iterative calculations respectively. But the principle of measurement is based on the numerical analysis of linear heat conduction, and the condition of heating rate of the specimens must be determined such that the linear principle may be valid even for the case that the thermal properties of the specimens vary strongly with temperature. The rate of the temperature rise and the time step of the iterative calculations are chosen suitably from the simulations of the measurements by a computer. An apparatus for measurement and examples of the measurements are shown in order to confirm the above principle of measurement experimentally. In the experiments soda-glass is chosen as the standard specimens because of its stable thermal properties, and polyethylene and polymethylmethacrylate are chosen as typical specimens because the thermal properties of polyethylene vary strongly with temperature between 0 °C and 100 °C and ones of polymethylmethacrylate do not. The results by this method are compared with our previous method^(1,6) of measurement. This method is shown to have the above mentioned merits and to be one of the useful methods of simultaneous measurement to determine the thermal properties as functions of temperature easily in short measuring time.

2. The principle of measurement

In Fig.2, a test specimen ② is placed between two standard specimens ① of which the thermal properties (thermal conductivity λ_s and thermal diffusivity α_s) are known. The specimens are heated from both the surfaces ($x=\pm L$) so that the temperatures at $x=\pm L$ may be nearly equal and rise

smoothly with time. The iteration method is applied to this system.

The procedures of the iterative calculations of this method are illustrated in a flow chart of Fig.3. The calculations in Fig.3 are composed of the following two groups: (A) the calculations to determine the thermal diffusivity by iterative calculations and to obtain the temperature distribution in the test specimen, and (B) the calculations of the amount of heat flux from two standard specimens into the test specimen to determine the heat capacity. These procedures of the calculations (A-1,2 and B-1,2,3 in Fig.3) will be shown as follows. In Fig.2 (b), the experimental temperatures at $x=\pm L$, $\pm l$ and 0 are denoted as $\theta_{\pm L}$, $\theta_{\pm l}$, θ_0 respectively, these temperatures are measured for every time interval of Δt and the thermal properties are determined for every time interval of Δt^* . These procedures are repeated and the temperature of the specimens is raised continuously to cover the necessary temperature range.

(A-1): The thermal diffusivity of the specimen is assumed to have certain value, and the temperature response U (defined by Eq.(4)), at the center surface of specimen

Fig.3 Flow chart of the principle of measurement

($x=0$) and the average temperature rise $\Delta\theta$ (defined by Eq.(5)), in the test specimen from time t_j to $t_{j+1}=t_j+\Delta\tau^*$, are calculated.

(A-2): As shown in Fig.4, the calculated value U of the temperature response at $x=0$ is compared with the experimental value u (defined by Eq.(6)). If both do not agree with each other with sufficient accuracy, the thermal diffusivity is reassumed to have another value and the above calculations are iterated. Depending on the sign of the difference of the temperature responses, u and U , the reassumed value of the thermal diffusivity a is determined as shown in Fig.4. As the result of these iterative calculations, the thermal diffusivity is determined. When the specimen is heated continuously from both the surfaces, the temperature response U is a monotonously decreasing function of the assumed value of thermal diffusivity a . In a special case that the temperatures $\theta_{\pm l}$ rise in proportion to time, U is in inverse proportion to a . These relations are convenient to the iterative calculations.

(B-1): The temperature distributions in the standard specimens are eliminated and from these results the amount of heat Q (obtained by Eq.(11)) flowing into the test specimen in $\Delta\tau^*$ (between t_j and t_{j+1}) is calculated.

(B-2): The heat capacity c_p is determined by substituting $\Delta\theta$ and Q obtained in the above into Eq.(12).

(B-3): The thermal conductivity λ is determined by the following equation, $\lambda = a \times c_p$.

The above calculations are iterated for every time interval $\Delta\tau^*$, and the thermal properties are determined through the necessary temperature range.

Now the practical way of the above calculations will be shown as follows.

(A-1) The calculations of the temperature responses and the temperature rises $\Delta\theta$ in the test specimen.

The time interval $\Delta\tau^*$ is chosen so short that the thermal properties may be considered to be constant, and then the temperature distributions in the test specimen are calculated by the explicit method of the finite difference equations based on linear heat conduction. The conditions of heating under which the solutions of the linear equations should hold good will be discussed in the next chapter.

The temperature distributions in the test specimen ($-l \leq x \leq l$, in Fig.2(b)) are calculated as follows.

The test specimen ($-l \leq x \leq l$) is divided into N nodes spaced equally and

Fig.4 Method of determining thermal diffusivity by iterative calculations

Table 3 Nomenclature

Nomenclature	Quantity	Dimension	Nomenclature	Quantity	Dimension
a	thermal diffusivity	m^2/hr	U	calculated temperature response (Eq.(4))	$^{\circ}C$
B	rate of heating	$^{\circ}C/hr$	x	co-ordinate	m
c	specific heat	$kcal/kg^{\circ}C$	Δx	divided space	m
$f_j(x)$	calculated temperature distribution at the time t_j	$^{\circ}C$	α	temperature coefficient of thermal conductivity	$1/^{\circ}C$
l, L	thickness, length	m	γ	temperature coefficient of thermal diffusivity	$1/^{\circ}C$
M	number of divisions of standard specimen		θ	temperature (mainly experimental temperature)	$^{\circ}C$
N	number of divisions of test specimen		θ_n^P	temperature in finite difference equation	$^{\circ}C$
q	heat flux	$kcal/m^2hr$	ϵ	relative error (Eq.(16))	
Q	amount of heat per unit area	$kcal/m^2$	λ	thermal conductivity	$kcal/mhr^{\circ}C$
$2R$	transverse length of the specimens	m	ρ	specific weight	kg/m^3
t	time	hr	$\Delta\tau$	time interval of measuring temperatures	hr
$t_j = j\Delta\tau^*$	time when the thermal properties are determined	hr	$\Delta\tau^*$	time interval of determining the thermal properties	hr
Δt	time step	hr	$\phi(t)$	temperature rise on each surface of the specimens (Eq.(3))	$^{\circ}C$
$T = \theta/(B l^2/Q_0)$	non-dimensional temperature				
u	experimental temperature response (Eq.(6))	$^{\circ}C$			

Suffix s : standard specimen, $0, \pm l, \pm L$: $x=0, \pm l, \pm L$

the temperature of the n th node ($x_n = -l + (n-1)\Delta x$, $n=1, 2, \dots, N+1$, $\Delta x = 2l/N$) at $t = t_j + P\Delta t$ is denoted as θ_n^P , where $t_j = j\Delta t^*$ ($j=0, 1, 2, \dots$). The thermal properties of the test specimen are considered to be constant because Δt^* is short and the temperature rise in the test specimen from t_j to t_{j+1} is small. When the thermal diffusivity is assumed to have certain value α , the fundamental finite difference equation is written as

$$\theta_n^{P+1} = \theta_n^P + \frac{\alpha \Delta t}{(\Delta x)^2} (\theta_{n-1}^P - 2\theta_n^P + \theta_{n+1}^P) \quad \left. \begin{matrix} (t_j \leq t \leq t_{j+1}, -l \leq x \leq l) \end{matrix} \right\} \dots (1)$$

The evaluated temperature distribution in the test specimen at $t = t_j$ is used as the initial condition, that is, when $t = t_j$ ($P=0$),

$$\begin{aligned} \theta_n^0 &= f_j(x_n), \quad (n=1, 2, \dots, N+1, \\ j &= 0, 1, 2, \dots) \end{aligned} \quad (2)$$

When $t = t_0 = 0$ ($j=0$), $f_0(x_n)$ expresses the temperature at start of heating which may be obtained experimentally, that is $f_0(x_n) = \theta_{t=0}$.

The temperatures $\phi_{\pm l}(t)$ which are functions of time are used as the boundary conditions at $x = \pm l$. The function $\phi_l(t)$ between $i\Delta t$ and $(i+1)\Delta t$ is approximated by assuming a linear variation. Thus

$$\begin{aligned} \varphi_i(t) &= \theta_l(i\Delta t) + [\theta_l((i+1)\Delta t) - \theta_l(i\Delta t)] \\ &\quad \times (t - i\Delta t) / \Delta t, \quad \{i\Delta t \leq t < (i+1)\Delta t, \\ i &= 0, 1, 2, \dots\} \end{aligned} \quad (3)$$

Where $\theta_l(i\Delta t)$ is the experimental temperature at $x=l$ and $t=i\Delta t$. The temperature $\phi_{-l}(t)$ at $x=-l$ is given in a similar fashion to Eq.(3).

From Eqs.(1)~(3), the temperature distribution $f_{j+1}(x_n)$, ($n=1, 2, \dots, N+1$) at $t = t_{j+1}$ is calculated and the temperature response U at $x=0$ is obtained as

$$U \equiv \{\varphi_{-l}(t_{j+1}) + \varphi_l(t_{j+1})\} / 2 - f_{j+1}(0) \quad \dots (4)$$

The average temperature rise in the test specimen from t_j to t_{j+1} is calculated by the following equation,

$$\begin{aligned} \Delta \theta &= \frac{1}{2l} \int_{-l}^l \{f_{j+1}(x) - f_j(x)\} dx \\ &= \frac{1}{2} \{f_{j+1}(x_1) - f_j(x_1) + f_{j+1}(x_{N+1}) \\ &\quad - f_j(x_{N+1})\} + \frac{1}{N-1} \sum_{n=2}^N \{f_{j+1}(x_n) - f_j(x_n)\} \end{aligned} \quad (5)$$

(A-2) Determination of thermal diffusivity α by iterative calculations.

The calculated temperature response U (defined by Eq.(4)) decreases monotonously with the thermal diffusivity α . As shown in Figs.3 and 4, the calculated temperature response U at $t = t_j$ is compared with the experimental one u at same time, where u is defined as

$$u \equiv \{\varphi_{-l}(t_{j+1}) + \varphi_l(t_{j+1})\} / 2 - \theta_0 \quad \dots (6)$$

From this result, each of $\alpha_1, \alpha_2, \dots$, and α_k is obtained in turn. α_k which satisfies the condition that $|a_{k-1} - \alpha_k| / \alpha_k < 1\%$, is considered to be a valid thermal diffusivity

α at $\theta = \{\phi_l(t_{j+1}) + \phi_{-l}(t_j)\} / 2$. Then the thermal diffusivity is determined. Simultaneously the temperature rise in the test specimen $\Delta \theta$ is calculated by Eq.(5).

(B-1) Calculations of the amount of heat conducted into the test specimen.

From the results of the numerical analysis of heat conduction in the standard specimens, the amount of heat conducted into the test specimen will be obtained as follows. The region $l \leq x \leq L$ in Fig.2(a) is divided into equally spaced M nodes just as in (A-1), and the temperature of the n th node ($x = l + (n-1)\Delta x_s$, $n=1, 2, \dots, M+1$, $\Delta x_s = (L-l)/M$) at $t = P\Delta t$ ($P=0, 1, 2, \dots$) is written as θ_{sn}^P . When the thermal properties of the standard specimen are known, the finite difference equation of nonlinear heat conduction is written as

$$\begin{aligned} \theta_{sn}^{P+1} &= \theta_{sn}^P + \frac{a_{sn} \Delta t}{(\Delta x_s)^2} \left\{ \frac{\lambda_{sn}(n-1, n)}{\lambda_{sn}} \right. \\ &\quad \times (\theta_{sn-1}^P - \theta_{sn}^P) + \frac{\lambda_{sn}(n, n+1)}{\lambda_{sn}} \\ &\quad \times (\theta_{sn}^P - \theta_{sn+1}^P) \left. \right\}, \quad (n=2, 3, \dots, M) \end{aligned} \quad (7)$$

The initial condition is

$$t=0 \quad (P=0), \quad \theta_{sn}^0 = \theta_{t=0} \quad (n=1, 2, \dots, M+1) \quad (8)$$

The boundary conditions are

$$\begin{aligned} x=l \quad (n=1), \quad \theta_{s1}^P &= \varphi_l(P\Delta t) \\ x=L \quad (n=M+1), \quad \theta_{sM+1}^P &= \varphi_L(P\Delta t) \end{aligned} \quad (9)$$

where $\phi_L(t)$ is obtained from the experimental temperature θ_L just as in Eq.(3), and λ_{sn} and a_{sn} are thermal conductivity and thermal diffusivity of the standard specimen at θ_{sn}^P respectively and

$$\lambda_s(n, n+1) \equiv \frac{1}{\theta_{sn+1}^P - \theta_{sn}^P} \int_{\theta_{sn}^P}^{\theta_{sn+1}^P} \lambda_s d\theta$$

The amount of heat $q_l(t)\Delta t$ per unit area which is conducted into the test specimen across the surface at $x=l$ between $t = P\Delta t$ and $t = (P+1)\Delta t$ is obtained by the following equation,

$$\begin{aligned} q_l(t) \Delta t &= -\frac{1}{2} \Delta x_s (c\rho)_{s1} (\theta_{s1}^{P+1} - \theta_{s1}^P) \\ &\quad - \lambda_s(1, 2) \frac{\theta_{s1}^P - \theta_{s2}^P}{\Delta x_s} \Delta t \quad \dots (10) \end{aligned}$$

where $(c\rho)_{s1}$ is the heat capacity of the standard specimen at the temperature θ_{s1}^P .

The amount of heat $q_{-l}\Delta t$ conducted into the test specimen across the surface at $x=-l$ is obtained just as $q_l\Delta t$. Then the amount of heat per unit area Q which is conducted into the test specimen across both the surfaces at $x = \pm l$ between $t = t_j$ and $t = t_{j+1} = t_j + \Delta t^*$ is obtained from the following equation,

$$\begin{aligned} Q &= \int_{t_j}^{t_{j+1}} \{q_l(t) + q_{-l}(t)\} dt \\ &= \sum_{P=1}^{j+1} \{q_l(t_j + P\Delta t) + q_{-l}(t_j + P\Delta t)\} \Delta t \quad \dots (11) \end{aligned}$$

(B-2) Determination of heat capacity.

From the temperature rise of the test specimen $\Delta \theta$ (Eq.(5)) and the amount of

heat conducted into the test specimen Q (Eq.(11)) in time $\Delta\tau^*$, the heat capacity $(c\rho)$ is obtained as follows,

$$c\rho = Q/(2l\Delta\theta) \dots\dots\dots(12)$$

(B-3) Determination of thermal conductivity.

From the thermal diffusivity a and the heat capacity $(c\rho)$ which were determined in (A-2) and (B-2) respectively, the thermal conductivity is obtained as

$$\lambda = a \cdot c\rho \dots\dots\dots(13)$$

Thermal properties obtained above are values between t_j and t_{j+1} , and then the thermal properties over the necessary temperature range of the measurement are obtained one after another by replacing t_j with t_{j+1} .

3. Determination of the conditions of the measurement

The necessary conditions of measurement — the sizes of the specimens, the rate of temperature rise and the time interval to determine the thermal properties — and the calculating conditions of data processing under which the above principles of measurement hold, are described following our previous papers⁽⁹⁾⁽¹⁰⁾ introducing the method by continuous heating.

3.1 Determination of conditions of heating rate under which the influence of the temperature dependencies of thermal properties can be neglected.

This method of measurement is investigated for the object to handle the specimens of which the thermal properties depend on temperature. But the thermal properties of the specimen are assumed to be constant within the temperature rise $\Delta\theta$ in $\Delta\tau^*$, and the principle of measurement is based on the finite difference equation (1) of linear heat conduction.

$$\lambda = \lambda_0(1 + \alpha T), \quad a = a_0(1 + \gamma T), \quad T = \theta / (Bl^2/a_0), \\ \theta_{\pm l} = Bt, \quad \Delta\tau_0 = 0.2l^2/a_0, \\ \epsilon_a: \text{Eq. (15)}, \quad \epsilon: \text{Eq. (16)}$$

Fig.5 Influence of the temperature dependencies of thermal properties ($\epsilon_a - \epsilon$)

The conditions — the rate of heating B and time interval $\Delta\tau^*$ — are determined from the simulations by computer just as in our previous methods⁽⁹⁾⁽¹⁰⁾ of measurement by continuous heating so that the influences of the temperature dependencies of the thermal properties can be neglected.

The thermal conductivity and the thermal diffusivity of the test specimen are considered to be linear functions of temperature represented as $\lambda(\theta) = \lambda_0(1 + \alpha\theta)$ and $a(\theta) = a_0(1 + \gamma\theta)$ respectively, and the temperatures $\theta_{\pm l}$ of the test specimen are considered to rise linearly with time ($\theta_{\pm l} = Bt$). First the temperature responses in the specimen are calculated by the finite difference equations of nonlinear heat conduction, and these calculated results are considered to be experimental ones. The thermal properties are determined according to the principle of measurement (mentioned in previous chapter) which is based on the solution of linear heat conduction, and these thermal properties are considered to be the results obtained experimentally and the thermal diffusivity and the heat capacity are denoted as a^* and $(c\rho)^*$ respectively. On the other hand, the thermal properties at the average temperature between t_j and t_{j+1} are considered to be the values which are sought, and the thermal diffusivity and the heat capacity are respectively defined as follows,

$$\left. \begin{aligned} \overline{(c\rho)} &= \frac{(c\rho)\{\varphi_i(t_j)\} + (c\rho)\{\varphi_i(t_{j+1})\}}{2} \\ \bar{a} &= \frac{a\{\varphi_i(t_j)\} + a\{\varphi_i(t_{j+1})\}}{2} \end{aligned} \right\} \dots\dots\dots(14)$$

The influences of the temperature dependencies of the thermal properties can be expressed by such relative deviations of thermal diffusivity and heat capacity as

$$\epsilon_a = (a^* - \bar{a})/\bar{a}, \quad \epsilon_{c\rho} = \{(c\rho)^* - \overline{(c\rho)}\}/\overline{(c\rho)} \dots\dots\dots(15)$$

$$\lambda = \lambda_0(1 + \alpha T), \quad a = a_0(1 + \gamma T), \quad T = \theta / (Bl^2/a_0), \\ \theta_{\pm l} = Bt, \quad \Delta\tau_0 = 0.2l^2/a_0, \\ \epsilon_{c\rho}: \text{Eq. (15)}, \quad \epsilon: \text{Eq. (16)}$$

Fig.6 Influence of the temperature dependencies of thermal properties ($\epsilon_{c\rho} - \epsilon$)

The rate of variation of heat capacity in time $\Delta\tau^*$ is denoted as

$$\varepsilon = \frac{(c\rho)\{\varphi_i(t_{j+1})\} - (c\rho)\{\varphi_i(t_j)\}}{(c\rho)} \dots\dots(16)$$

and from the results calculated by using the various values of the coefficients of the thermal properties, α and γ , the relations $\varepsilon_\alpha - \varepsilon$ and $\varepsilon_{c\rho} - \varepsilon$ are obtained as in Figs.5 and 6 respectively. From Figs.5 and 6, it can be seen that the influences of the temperature dependencies of the thermal properties can be neglected when $|\varepsilon|$ is small.

Therefore the rate of heating B and the time interval $\Delta\tau^*$ should be determined such that the following relation may be obtained,

$$|\varepsilon| < 2 \sim 3\% \dots\dots\dots(17)$$

When the thermal properties of a specimen vary strongly with temperature, for example when they are expressed as $(c\rho)(\theta) = (c\rho)_0(1 + \theta/100)$, the value of $B\Delta\tau^*$ should be determined by Eqs.(16) and (17) as $B\Delta\tau^*$ ($\neq \Delta\theta$) $\leq 2^\circ\text{C}$. If the values of the temperature coefficients of the thermal properties, α and γ , can not be predicted, the thermal properties may be measured with an appropriate rate of heating (for example $B\Delta\tau^* = 2^\circ\text{C}$) and it should be confirmed whether the results of measurement satisfy Eq.(17) or not. If the results do not satisfy Eq.(17), the thermal properties should be measured again at a decreased heating rate.

As shown in our previous paper⁽⁹⁾ in the method of measurement by continuous heating, the consequences of the above discussions are also valid even when the thermal properties are not expressed by the linear functions of temperature or the temperature of the specimen does not rise linearly with time.

3.2 Determination of the size of the specimens

As shown in our previous paper⁽⁹⁾, the influence of heat loss from the side surfaces of the specimens can be neglected if the ratio $2R/L$ satisfies the following relation,

$$2R/L \geq 12 \sim 14 \dots\dots\dots(18)$$

where $2R$ denotes the transverse length of the specimens.

3.3 Determination of the conditions of data processing computations

The principle of measurement is based on the finite difference equations of explicit method. Therefore space and time must be divided adequately so that sufficient accuracy may be obtained and the computation time may be short. The reference (18) has shown that a stable solution whose maximum value of error is less than $2\sqrt{3}\%$ can be obtained under the following

conditions,

$$\frac{a\Delta t}{(\Delta x)^2} \leq \frac{1}{2}, \quad \frac{\Delta\tau^*}{\Delta t} > 3, \quad \frac{a\Delta\tau^*}{(\Delta x)^2} > 0.4$$

If $a\Delta t/(\Delta x)^2$ is maintained constant and the number of divisions of space is doubled, the error of the computation will be decreased by one-fourth. Then the conditions under which the maximum value of error becomes less than 1% are written as

$$\frac{a\Delta t}{(\Delta x)^2} \leq \frac{1}{2}, \quad \frac{\Delta\tau^*}{\Delta t} > 12, \quad \frac{a\Delta\tau^*}{(\Delta x)^2} > 1.6 \dots\dots\dots(19)$$

In order to shorten the computation time, both space and time should be divided such that the product $\Delta x \times \Delta t$ may be the largest within the conditions of Eq.(19).

4. Experiments

In this chapter, the apparatus of measurement according to the above principles and the conditions investigated in the foregoing chapter, and the results of examples of the experiment are described. The kinds of the specimen are arbitrary in principle, but the thermal properties of the standard specimen should be stable under heating. Soda-glass is chosen as the standard specimen and its thermal properties are determined precisely by the method of absolute measurement by step-wise heating as follows, $\lambda_s = 0.87(1 + 0.0007 \times \theta)$, $\alpha_s = 17.5 \times 10^{-4}(1 - 0.0010\theta)$. As the test specimens, polyethylene ($\rho = 957$) and polymethylmethacrylate ($\rho = 1190$) are chosen, because the thermal properties of polyethylene vary strongly with temperature within a narrow temperature range and those of polymethylmethacrylate do not. The experimental results obtained by the present method of measurement are compared with those by the method of absolute measurement by step-wise heating and because both the results agree well with each other, this method of measurement is found to be useful.

4.1 Apparatus of measurement and experimental procedures

Both the thicknesses of the test specimen and the standard specimen l , l_s are chosen to be 5 mm. The transverse length of the specimens $2R$ is determined to be 150 mm so that Eq.(18) should be

Fig.7 Apparatus of measurement

satisfied, where $L=l+l_s=10$ mm. The apparatus of measurement is illustrated in Fig.7. In Fig.7, the test specimen ② (polyethylene or polymethylmethacrylate) is placed between two standard specimens ① (soda-glass), and these specimens are also placed between two copper plates of 3 mm thick so that heat conduction in the specimens can be regarded as one-dimensional. Heaters are made of graft carbon cloth and are heated by a program controller so that the temperatures of the specimens may rise linearly with time. Glass of 10 mm thickness is placed between heater and copper plate so that the temperatures of the specimens may rise smoothly. Insulators made of asbestos of about 30 mm thickness are set outside the heaters and all of them bolted up between two steel plates. The apparatus of measurement is set in a vacuum vessel which is evacuated to 10^{-3} mmHg in order to decrease heat loss from the side surfaces of the specimens. Silicone grease is used to minimize the thermal resistances on each surface of the specimens. The temperatures are measured by 0.1 mmφ copper-constantan thermocouples and recorded by a digital voltmeter. The thermocouples are adhered in the grooves of 0.2~0.3 mm depth which are hollowed out on the surfaces of the specimens with a diamond cutter. The depth of the grooves is measured with a dialgage. Seven temperatures, θ_{s+l} and θ_{s+l} in the standard specimens and θ_0 and θ_{+l} in the test specimen, are measured.

The experiments are made within the range from 0°C to 100°C by considering the softening point of polyethylene. The rate of heating B is determined as 2 or 4°C/min and the temperatures are measured every Δt ($\Delta t=1$ or 0.5 minutes). If the time interval Δt^* is determined as $\Delta t^*=3\Delta t=3$ or 1.5 minutes, the temperature rise of the specimen $\Delta\theta$ in Δt^* is deduced as $B\Delta t^*\approx 6^\circ\text{C}$. The influence of the temperature dependencies of the thermal properties are considered to be negligible because ϵ in Eq.(16) is eliminated as $|\epsilon|\approx 3\%$ from the results of measurement in next chapter.

The conditions of the computations are determined such that Eq.(19) may be satisfied. The test specimen ($-\ell \leq x \leq \ell$) is divided into eight nodes ($N=8$) and the standard specimens ($\ell \leq x \leq L$, $-L \leq x \leq -\ell$) are divided into four nodes ($M=4$). The time step Δt is chosen such that the relations $a\Delta t/(\Delta x)^2 \div 0.4$, $\alpha_s \Delta t/(\Delta x_s)^2 \div 0.4$ and $\Delta \tau^*/\Delta t > 16$ may be satisfied.

4.2 Results of measurements

The results of measurements are shown in Figs. 8-14. Figure 8 shows the temperature rise and the temperature differences in the specimens heated at a heating rate of 4 °C/min, where the specimen is polyethylene. In this measurement, the initial temperature was 4.7 °C, the maximum temperature was 100 °C, and the thermal properties were determined every 1.5 minutes. It took 30 minutes to complete this measurement. Data processing time to obtain the results was about one minute by

Fig.8 Temperature rise and temperature difference in the specimens

$$(\ell_s = 4.90, 4.89 \text{ mm}, \ell = 4.84, 4.84 \text{ mm})$$

Fig.9 Experimental results of thermal conductivity of polyethylene

Fig.10 Experimental results of thermal diffusivity of polyethylene

Fig.11 Experimental results of heat capacity of polyethylene

($l_s=4.90, 4.93$ mm, $l=4.89, 4.85$ mm)

Fig.12 Experimental results of thermal conductivity of polymethylmethacrylate

Fig.13 Experimental results of thermal diffusivity of polymethylmethacrylate

HITAC 8700 at the Computer Center of Tokyo Institute of Technology. The results of measurements are shown by Experiment No.3 in Figs.9~11. In Figs.9~11, the results of polyethylene in the case that the rate of heating is 2 °C/min are also shown, and the experimental results of the same specimen by the method⁽¹⁶⁾ of absolute measurement by step-wise heating are also shown. The results of polymethylmethacrylate are also shown in Figs.12~14.

4.3 Discussion on the results

As shown in Fig.8, the maximum temperature difference in the test specimen is less than 5 °C, the difference between the temperatures on both the surfaces ($x=\pm L$) is less than 2°C, and the temperatures of the specimens are so controlled as to rise linearly with time. Therefore, the heating can be considered as properly satisfying the conditions in Chap.3. As shown in Figs.9~14, the experimental results of polyethylene that vary much with temperature and those of polymethylmethacrylate that vary little with temperature coincide with each other within the range of about 5 % at any temperature, and still do so with the experimental results of the same specimens by the method⁽¹⁶⁾ of absolute measurement by step-wise heating within the same range. Using the apparatus of measurement and the experimental procedures which satisfy the conditions determined in Chap.3, the principle of measurement holds good. And this method of measurement is found to be useful to determine the thermal properties as functions of temperature over a wide temperature range. By this method, the thermal properties are measured within 0.5~1 hour over the temperature range of 100 °C. On the other hand, when the thermal properties are measured six times every 20 °C by the usual method of transient simultaneous measurement by step-wise heating, the

Fig.14 Experimental results of heat capacity of polymethylmethacrylate

measurement takes more than 24 hours because long time is needed to get the initial conditions. Therefore this method is one of those that can determine the temperature dependencies of the thermal properties in short time. In this method, the thermal properties of the standard specimen must be known and the calculations to determine the thermal properties must be carried out by a computer. If these necessary preparations are made, this method has the following merits — it is not necessary to measure the amount of heat generated by heaters and the way of heating of the specimens is arbitrary as far as the temperatures of the specimens are raised smoothly with time — and this method can determine the thermal properties more easily than our previous method⁽¹⁰⁾ of simultaneous measurement by continuous heating. This method is found to be able to determine easily the thermal properties by adjusting the heating rate B and the time interval $\Delta\tau^*$ when it is applied to any case that the thermal properties of specimens vary continuously and smoothly with temperature except the case that the thermal properties (near the temperature of phase transition) vary uncontinuously.

5. Conclusions

In the present paper, principle of the method of measuring properties by iterative calculations is presented, and is applied to the method of simultaneous measurement of the thermal properties which are essential for the analysis of nonlinear heat conduction problems. This method of measurement is improved by combining our previous method of transient measurement by continuous heating with the transient comparison method with standard specimen, and the method of iterative calculations based on numerical analysis is applied to the principle of measurement. Above principle of this method is illustrated in Figs. 2 and 3.

The necessary conditions of measurement and of calculations are determined theoretically in Eqs. (16)~(19) so that the principle of measurement may hold good.

To verify this method, experiments are made, where thermally stable soda-glass is used as standard specimens, and polyethylene of which the thermal properties vary strongly with temperature and polymethylmethacrylate of which the thermal properties vary only a little are used as test specimens. The results of these measurements are compared with the results obtained by the method of measurement by

step-wise heating, the same specimens being used in both the measurements. Both the results coincide well with each other.

The present method of measurement can be considered to be one of the useful methods of measurement which can simultaneously determine the thermal properties — thermal conductivity, thermal diffusivity and heat capacity — as functions of temperature.

By the way, it seems that the principle of measurement based on such iterative calculations as presented in this paper can be applied to methods of measuring properties in various engineering fields.

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