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Thermal Conductivity Measurement of Semitransparent Solids by Hot-Wire Technique[1]

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A new simple analytic model applicable to the measurement system of a hot wire and a semitransparent solid material is developed. An experimental study is carried out on a special glass sample, glass K9, in the temperature range of 297 to 1230 K, and the radiation-free thermal conductivity is reported.

KEY WORDS: heat radiation; hot wire; thermal conductivity; semitransparent material.

1. INTRODUCTION

Semitransparent materials, such as glass and plastics, are partly transparent to heat radiation. They absorb and emit radiant energy, and are also called radiation-participant materials. The transient hot-wire technique is widely accepted as the most precise method for the measurement of the thermal conductivity of fluids. It has been extended to various domains such as composite materials, wet porous media, and solids at high temperatures. However, if it is used directly for the measurement of the thermal conductivity of semitransparent solid materials, the result is influenced by radiation. We have studied numerically the transient coupled conductive–radiative heat transfer inside semitransparent materials using the hot-wire technique [1]. The influences of thermal radiation on the inner heat flux of the sample and on the temperature rise of the hot wire were analyzed. Two new concepts, the effective mean absorption coefficient of the material and the effective emissivity of the hot-wire surface, were introduced; thus,

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the governing equation and the relevant contact boundary condition were simplified.

Based on [1], a new simple analytic model applicable to the hot-wire measurement system and a semitransparent solid material is derived in the present paper by means of the Laplace transform and thermal quadrupole method [2–4]. Based on parameter estimation, sensitivity analyses are made for the simplified model. An experimental study is carried out on a special glass sample, glass K9, in the temperature range of 297 to 1230 K.

2. MATHEMATICAL DESCRIPTION

The ideal model of the transient hot-wire instrument consists of an infinitely long thin cylinder (hot wire) heated by a constant, uniform internal source and located in an semitransparent, infinite cylindrical solid material (sample) which can absorb and emit radiation. A thermal contact resistance R_c exists between the hot wire and the sample. The hot wire and sample with constant thermophysical properties are initially at a uniform temperature T_0. From time $t=0$, the whole system is heated and the temperature response of the hot wire is measured and recorded simultaneously; thus, the thermophysical properties of the sample can be estimated.

It is assumed that the temperature of the sample varies only with radial position r and time t, and, furthermore, the hot-wire surface A_1 is gray, the sample outer surface A_2 is black, and the middle semitransparent material is assumed to be isotropic, gray, and nonscattering. In a cylindrical coordinate system, we obtain the energy equation of the sample ($r_1 < r < r_2$ and $t > 0$) as follows:

$$(\rho c_p)\frac{\partial T}{\partial t} = \frac{\lambda}{r}\frac{\partial}{\partial r}\left(r\frac{\partial T}{\partial r}\right) + q_R \tag{1}$$

where $q_R = Q'_{V \to dV_i} + Q'_{A_1 \to dV_i} + Q'_{A_2 \to dV_i} - 4KE_i$. The relevant interface condition between the hot wire and sample ($r = r_1$ and $t > 0$) is

$$-\lambda(\partial T/\partial r)_1 = (T_s - T_1)/2\pi r_1 l R_c \tag{2}$$

Here, ρc_p and λ are the heat capacity per unit volume and the thermal conductivity of the sample, respectively, K is the mean absorption coefficient, T_s is the temperature of the hot-wire surface, T_1 is the temperature of the sample inner surface, and r_1, r_2, and l are the inner radius, outer radius, and the length of the sample, respectively. The subscript 1 denotes the sample. $Q'_{V \to dV_i}$, $Q'_{A_1 \to dV_i}$, and $Q'_{A_2 \to dV_i}$ represent the gradients of one-way radiation heat fluxes from the entire sample volume V, and the surfaces A_1 and A_2, respectively, to the volume element dV_i. Here $4KE_i$

represents the gradients of the total emitting heat flux from dV_i, and $E_i = n^2\sigma T^4$, where n is the refractive index of the sample and σ is the Stefan–Boltzmann constant.

For the hot wire, similarly, we write the following conduction equation ($0 < r < r_0$ and $t > 0$):

$$(\rho c_p)_s \frac{\partial T}{\partial t} = \lambda_s \frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial T}{\partial r}\right) + \dot{q} \tag{3}$$

and the corresponding boundary condition ($r = r_0$ and $t > 0$)

$$\lambda_s\left(\frac{\partial T}{\partial r}\right)_s = \lambda\left(\frac{\partial T}{\partial r}\right)_1 + \varepsilon(Q_{V\to dA_1} + Q_{A_2\to dA_1}) - \varepsilon n^2\sigma T_s^4 \tag{4}$$

Here, $(\rho c_p)_s$ and λ_s are the heat capacity per unit volume and the thermal conductivity of the hot wire, respectively, and ε is the emissivity of the hot-wire surface A_1. The subscript s denotes the hot wire. $Q_{V\to dA_1}$ and $Q_{A_2\to dA_1}$ represent the one-way radiation heat fluxes to an inner surface element dA_1 from the sample volume V and the outer surface A_2, respectively.

Obviously, it is very difficult to obtain an analytic solution to the above integro-differential equations. The present authors [1] developed a numerical method to solve the equations and study and simulate the combined conductive–radiative heat transfer. The results indicate that all of the radiative terms in Eq. (1) can be merged into one term by just considering the overall effect of emission and absorption. By introducing the concept of the effective mean absorption coefficient K_x and adopting the temperature rise variable $\bar{T} = T - T_0$, we can simplify Eq. (1) as follows:

$$\frac{1}{a}\frac{\partial \bar{T}}{\partial t} = \left(\frac{\partial^2 \bar{T}}{\partial r^2} + \frac{1}{r}\frac{\partial \bar{T}}{\partial r}\right) - \frac{16K_x n^2\sigma T_0^3}{\lambda}\bar{T} \tag{5}$$

Here, a is the thermal diffusivity of the sample and K_x obeys the condition $0 \leqslant K_x \leqslant K$.

In the same way, for the wire–sample boundary condition, Eq. (4), considering the overall effect of emission and absorption between the hot-wire surface and the sample, and furthermore introducing the concept of the effective emissivity of wire surface ε_x, we obtain the following simplified boundary condition:

$$-\lambda_s\left(\frac{\partial \bar{T}}{\partial r}\right)_s = \frac{\bar{T}_s - \bar{T}_1}{2\pi r_0 l R_c} + 4\varepsilon_x n^2\sigma T_0^3 \bar{T}_s \tag{6}$$

where ε_x obeys the condition $0 \leqslant \varepsilon_x \leqslant \varepsilon$.

The initial condition can be written as ($0 < r < r_2$ and $t = 0$)

$$\bar{T}(r, t) = 0 \tag{7}$$

3. TEMPERATURE RISE OF A HOT WIRE

The Laplace transform and thermal quadrupole methods [2] are used to solve the above heat transfer problem.

3.1. Quadrupole of a Semitransparent Cylindrical Sample

θ and ϕ are the Laplace transforms of the temperature rise $\bar{T}$ and heat flux Q, respectively. By virtue of Eq. (7), performing a Laplace transform on Eq. (5) and solving the transformed differential equations, we obtain the solution as [3, 4]:

$$\begin{pmatrix} \theta_1 \\ \phi_1 \end{pmatrix} = \begin{pmatrix} A & B \\ C & D \end{pmatrix} \begin{pmatrix} \theta_2 \\ \phi_2 \end{pmatrix} = [M] \begin{pmatrix} \theta_2 \\ \phi_2 \end{pmatrix} \tag{8}$$

where subscript 1 refers to r_1 and 2 refers to r_2. The matrix elements are:

$$A = \alpha_2 [I_0(\alpha_1) K_1(\alpha_2) + I_1(\alpha_2) K_0(\alpha_1)]$$

$$B = [I_0(\alpha_2) K_0(\alpha_1) - I_0(\alpha_1) K_0(\alpha_2)]/(2\pi\lambda l)$$

$$C = 2\pi\lambda l [I_1(\alpha_2) K_1(\alpha_1) - I_1(\alpha_1) K_1(\alpha_2)]$$

$$D = \alpha_1 [I_0(\alpha_2) K_1(\alpha_1) + I_1(\alpha_1) K_0(\alpha_2)]$$

where $\alpha_1 = (p/a + G)^{1/2} r_1$, $\alpha_2 = (p/a + G)^{1/2} r_2$, and $G = 16 K_x n^2 \sigma T_0^3/\lambda$. Here p is the Laplace variable. I_0, I_1, K_0, and K_1 are modified Bessel functions of the first and second kind and of order 0 and 1, respectively.

The linear relationship between input and output variables from Eq. (8) can be expressed as a quadrupole as shown in Fig. 1a. [M] is the inverse transmitting matrix, and the four elements obey the relation $AD - BC = 1$. Thus, this quadrupole is equivalent to the network of three impedances as shown in Fig. 1b, i.e.,

$$Z_1 = (A - 1)/C, \qquad Z_2 = (D - 1)/C, \qquad Z_3 = 1/C \tag{9}$$

When r_2 tends to infinity, we get the impedance of an infinite, semitransparent material as illustrated in Fig. 1c:

$$Z_1 \to Z = K_0(\alpha_1)/2\pi\lambda l \alpha_1 K_1(\alpha_1) \tag{10}$$

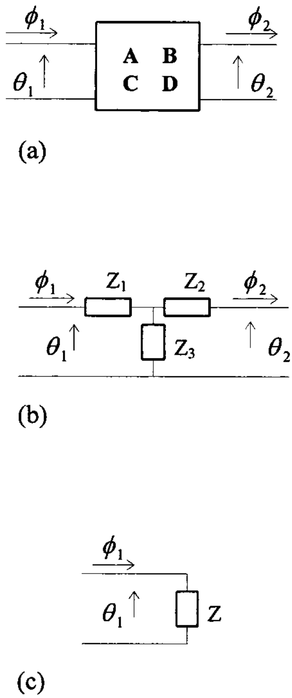

Fig. 1. Thermal quadrupole and impedances. (a) Thermal quadrupole. (b) Equivalent impedance. (c) Impedance of an infinite sample.

3.2. Hot Wire

The average temperature rise of the wire $\bar{T}_m$ and the heat flow Q_m are chosen as input variables; they are also the measured parameters:

$$\bar{T}_m(t) = 2r_0^{-2} \int_0^{r_0} \bar{T}(r, t) r \, dr, \qquad Q_m = \pi r_0^2 \, l\dot{q}(t)$$

θ_m and ϕ_m are the Laplace transforms. The same approach as before is used, and from Eqs. (3) and (7), we obtain:

$$\begin{bmatrix} \theta_m \\ \phi_m \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_0 \\ \phi_0 \end{bmatrix} \tag{11}$$

where the subscript 0 refers to r_0 and

$$A = 1$$
$$B = I_0(\alpha_0)/2\pi\lambda_s l\alpha_0 I_1(\alpha_0) - 1/(\rho c_p)_s V_s p = Z_s$$
$$C = (\rho c_p)_s V_s p = C_s p$$
$$D = \alpha_0 I_0(\alpha_0)/2I_1(\alpha_0)$$

and $\alpha_0 = \sqrt{p/a_s r_0}$, $V_s = \pi r_0^2 l$. Hence, the impedances corresponding to this quadrupole are

$$Z_1 = 0, \qquad Z_2 = Z_s, \qquad Z_3 = 1/C_s p \tag{12}$$

3.3. Contact Boundary Between Wire and Sample

We define in Eq. (6) that

$$R_x = 1/8\pi r_0 l\varepsilon_x n^2 \sigma T_0^3$$

which represents the thermal radiative resistance of the hot-wire surface. It is easy to show that the following solution conforms to Eqs. (2) and (6):

$$\begin{pmatrix}\theta_0\\ \phi_0\end{pmatrix} = \begin{pmatrix}A & B\\ C & D\end{pmatrix}\begin{pmatrix}\theta_1\\ \phi_1\end{pmatrix} \tag{13}$$

where $A = 1$, $B = R_c$, $C = 1/R_x$, $D = 1 + R_c/R_x$.

The corresponding impedances are

$$Z_1 = 0, \qquad Z_2 = R_c, \qquad Z_3 = R_x \tag{14}$$

3.4. Quadrupole of the Measurement System

When connecting the impedance networks of the hot wire, contact boundary, and infinite semitransparent sample in series, we use a quadrupole model of a hot-wire–semitransparent-material measurement system composed of impedances, as shown in Fig. 2.

The wire impedance Z_s becomes a pure resistance, $Z_s = 1/(8\pi\lambda_s l)$, for long enough time $t \gg r_0^2/a_s$, that is, $pr_0^2/a_s \ll 1$ in Laplace space. In the quadrupole model, C_s and R_s are the total heat capacity and thermal resistance of the hot wire, R_x is the thermal radiative resistance of the wire surface, R_c is the thermal contact resistance, and Z is the thermal impedance of the sample.

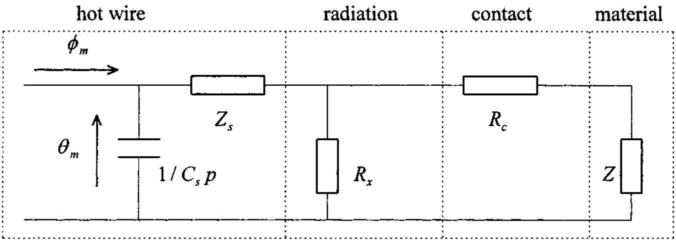

Fig. 2. The quadrupole of the wire–semitransparent-material measurement system.

With $r_1 = r_0$, the average temperature rise of the hot wire can then be found very simply in Laplace space:

$$\theta_m = \phi_m / \{ C_s p + [R_s + (R_x^{-1} + (R_c + Z)^{-1})^{-1}]^{-1} \} \tag{15}$$

Equation (15) is an analytic expression and designates the simplified model. Thus, the hot-wire average temperature rise $\overline{T}_m(t)$ is determined at any one time t by means of the numerical inversion of the Laplace transform [5].

4. PARAMETER ANALYSIS

The simplified model, Eq. (15), includes seven parameters:

$$\beta_1 = r_0^2/4a, \qquad \beta_2 = 1/4\pi\lambda l, \qquad \beta_3 = R_c, \qquad \beta_4 = C_s$$

$$\beta_5 = Gr_0^2/4, \qquad \beta_6 = R_s, \qquad \beta_7 = R_x$$

The sensitivity coefficient X_i of parameter β_i is defined as $\beta_i \, \partial \overline{T}_m / \partial \beta_i$ ($i = 1, 2, ..., 7$). Figure 3 shows typical variations of the sensitivity coefficient with time. A study of the sensitivities indicates that the two parameters β_1 and β_3 are correlated at $t < 10$ s, and β_2 and β_7 are correlated over the complete useful time range. It is therefore impossible to identify them simultaneously. On the other hand, the sensitivity coefficient of β_4 is so small for $t > 1$ s, and the sensitivity of β_6 is so small at all times that they cannot be estimated. Thus, according to the simplified model, Eq. (15), it is possible to only estimate the three parameters β_2, β_3, and β_5 simultaneously, that is, λ, R_c, and K_x.

In practice, it is easy to know the radius r_0, the length l, the thermal conductivity λ_s, the volumetric heat capacity $(\rho c_p)_s$, and the surface emissivity ε of the hot wire. The volumetric heat capacity ρc_p and the refractive index n of the sample can also be known by other methods. But

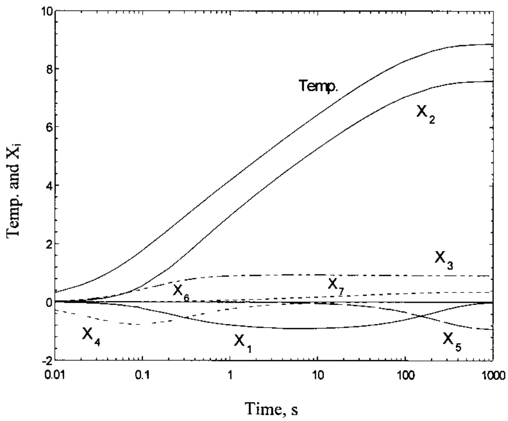

Fig. 3. Temperature rise of wire and sensitivity coefficients.

there is still an unknown coefficient ε_x in parameter β_7; its value must be known before estimating λ, R_c, and K_x. By virtue of $0 \leqslant \varepsilon_x \leqslant \varepsilon$, we generally let ε_x be equal to ε. The numerical simulations of parameter estimations demonstrate that a 0.7% to 2% relative error in estimated thermal conductivity will result from $\varepsilon_x = \varepsilon$, and this error can be eliminated through repeated iteration. First, let $\varepsilon_x = \varepsilon$; then the values of λ, R_c, and K_x are obtained by parameter estimation. Next, ε_x is corrected, and the parameter estimation is repeated. The iterative process does not end until a certain precision in thermal conductivity is attained. A more detailed calculation procedure is presented in [5].

5. EXPERIMENTAL STUDY

5.1. Experimental Setup

The experimental setup of the hot-wire method is presented in Fig. 4a. The sample ① is made up of two stacked, half-round cylindrical glass blocks (ϕ 105 mm $\times$ 210 mm). A cross-groove is cut into the lower half block in order to hold the hot wire and the thermocouple. The sample is placed in a special container, and a black coating is applied to the sample to make its outer surface a blackbody surface. Both the wire and the sample are set into a furnace ② whose temperature can be regulated between 20 and 1000°C.

The hot wire ③ is a FeCrAl alloy with a 0.95 mm diameter. A NiCr–NiSi thermocouple ④ of ϕ 0.5 mm is welded crosswise on the center of

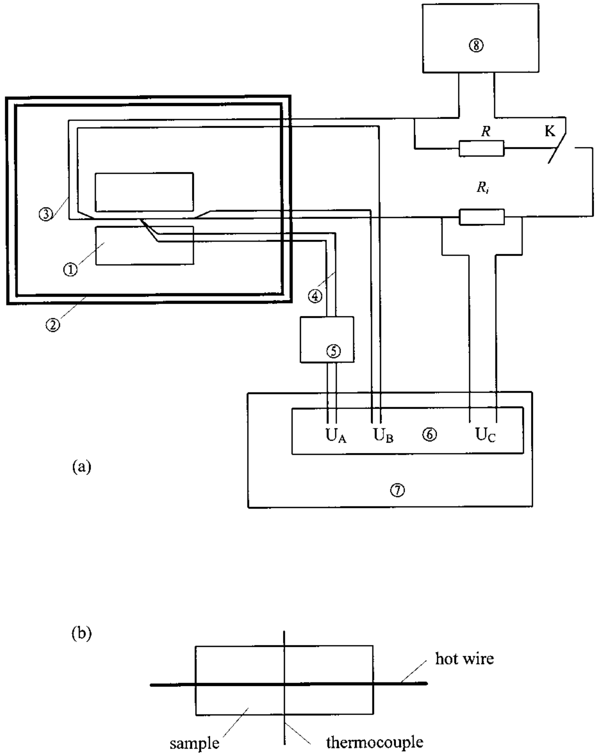

Fig. 4. Experimental setup and cross wire. (a) Experimental setup: ① sample, ② furnace, ③ hot wire, ④ thermocouple, ⑤ signal conditioning module, ⑥ A/D-converting board, ⑦ computer, ⑧ constant-current generator. (b) Cross wire.

the hot wire, as shown in Fig. 4b, and this structure is called the cross-wire. The signal conditioning module ⑤ amplifies the weak thermoelectric potential from the thermocouple to a 0 to 5 V signal U_A, which is inputted to an A/D converting board ⑥, and then to the computer ⑦. R_i is a standard resistance, and R is the secondary resistance.

A constant-current generator ⑧ allows a fixed current (0 to 2.5 A) to go through the wire. Before the measurements, the current goes through the secondary stabilization circuit. As soon as the switch is closed, the wire heats up and measurements start. The computer automatically records the voltage U_A, the voltage U_B across the wire, and the voltage U_C across the R_i simultaneously in order to determine the temperature rise and electric power consumption of the wire.

In the cross-wire scheme, the thermocouple attached to the hot wire will bring about additional loss of heat conduction and cause a local temperature drop at the cross joint of wire and thermocouple. If the thermophysical properties of the hot wire and thermocouple are almost the same, an approximate relation is obtained [5]:

$$\bar{T}_t(t)/\bar{T}_m(t) = 1/[1 + (r_t/r_0)^2] \tag{16}$$

where $\bar{T}_t(t)$ is the temperature rise of cross joint, i.e., the measured temperature rise of the hot wire, $\bar{T}_m(t)$ is the temperature rise of the hot wire without the attached thermocouple, and r_t/r_0 is the radius ratio of the thermocouple to the hot wire. It is obvious that the thinner the radius of thermocouple, the more closely does the measured temperature rise of the hot wire approach the true value. In the case of a thicker thermocouple, the measured temperature rise must be corrected according to Eq. (16) in order to achieve a reliable experimental result.

5.2. Experimental Results and Discussion

For the case of semitransparent materials at high temperatures, where the effect of radiative heat transfer is significant, the variation of the temperature rise of the hot wire $\bar{T}_m(t)$ with the logarithm of time $\ln(t)$ is no longer clearly linear in the latter portion, but is curved concave to the $\ln(t)$ axis. An experimental study on a glass sample, glass K9, verifies this relationship; the parameters employed for the experimental measurement are reported in Table I.

Figure 5 shows the variation of the temperature rise of the hot wire at a temperature of 297 K, the measured wire temperature rise $\bar{T}_m$ (dots), and the calculated temperature rise $\bar{T}_c$ (solid line) obtained with identified values of parameters. The lower curve in Fig. 5 is the residual curve

Table I. Parameters for the Experiment

Sample:	glass K9	
	$n = 1.5163$, $\rho = 2520\ \text{kg} \cdot \text{m}^{-3}$	
	$c_p = 0.8$ to $1.0\ \text{kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$	
Hot wire:	FeCr25Al5	
	$\lambda = 16.75\ \text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$,	$\rho = 7100\ \text{kg} \cdot \text{m}^{-3}$
	$c_p = 0.494\ \text{kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$,	$\varepsilon = 0.04$ to 0.1
	$q = 6\ \text{W} \cdot \text{m}^{-1}$ ($T < 1100$ K)	
	$q = 8\ \text{W} \cdot \text{m}^{-1}$ ($T > 1100$ K)	
Thermocouple:	NiCr–NiSi	

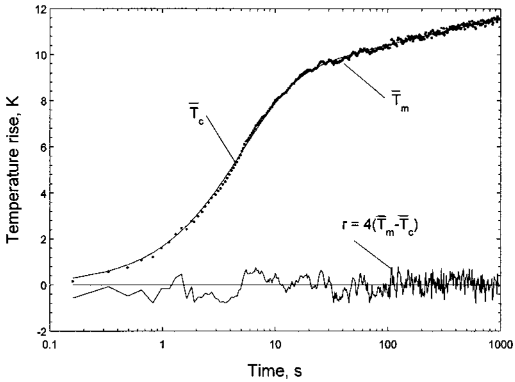

Fig. 5. Temperature rise of hot wire vs. time at $T = 297$ K.

$r = 4(\bar{T}_m - \bar{T}_c)$ showing the deviation between the measured and the calculated temperature rise. The experimental results indicate that the temperature rise $\bar{T}_m(t)$ with $\ln(t)$ is still linear in the latter portion when the temperature is below 678 K. Under these circumstances, the effect of heat radiation can be neglected and the sample is treated as a nontransparent material. Then, the thermal conductivity of the sample λ and the total thermal resistance R_t can be determined [3].

When the temperature is larger than 678 K, the change in temperature rise $\bar{T}_m(t)$ begins to become concave to the $\ln(t)$ axis, and becomes increasingly curved with an increase in temperature. Figure 6 shows the variation of the temperature rise of a hot wire at a temperature of 960 K; the solid line in Fig. 6a represents the calculated temperature rise without consideration of the radiative heat transfer and in accordance with the model for a nontransparent material. By observing the residual or deviation curve in Fig. 6a, a systematic curvature is evident, and this implies that the model does not fit the experimental data. The solid line in Fig. 6b represents the calculated temperature rise including the effect of heat radiation and in accordance with the new, simplified model, Eq. (15). It is observed that the residuals are well distributed and randomly oscillate around zero, and thus have the character of random error. This implies that the new model basically fits the experimental data. The new simplified model includes the effect of heat radiation on the heat transfer, and the parameters estimated for the new model are more realistic and reasonable than the parameters estimated for the old model.

Figure 7 presents the dependence of the thermal conductivity of glass K9 on temperature. Here the “squares” denote the results obtained

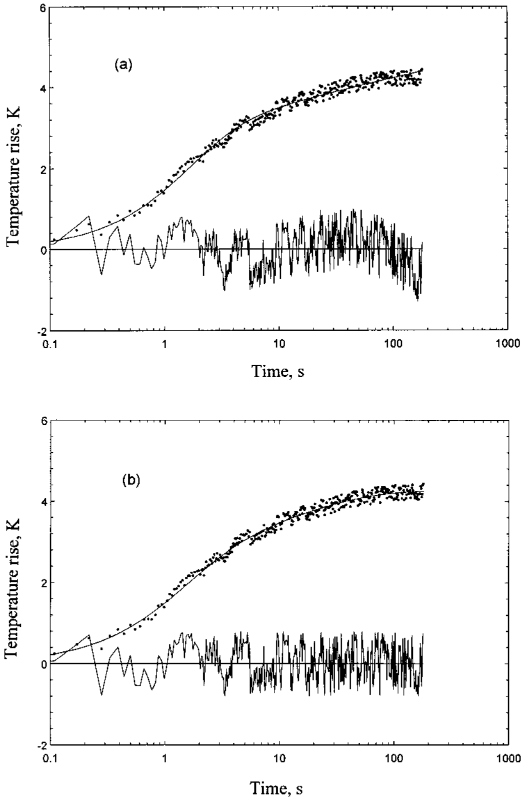

Fig. 6. Temperature rise of hot wire at $T = 960$ K (a) With the model of a nontransparent material. (b) With the new model of a semitransparent material.

without including the effect of radiation. The "circles" denote the results obtained including the effect of heat radiation inside only the sample and using the effective mean absorption coefficient K_x. The "triangles" denote the results obtained including the effects of radiation of both the wire and sample, and using both the effective mean absorption coefficient K_x and the effective emissivity of the hot-wire surface ε_x. The solid line corresponds to a least-squares fit of the experimental points.

According to the known composition of glass K9, we can determine the thermal conductivity at ambient temperature on the basis of some empirical formulas; $\lambda = 0.854\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ by the Russ formula [6]. This value is in good agreement with the result of $\lambda = 0.862\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ obtained in our experiment at a temperature of 297 K.

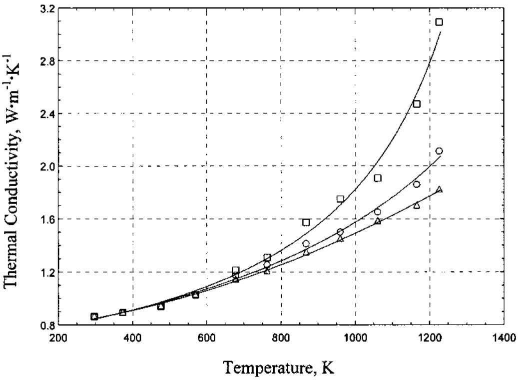

Fig. 7. Thermal conductivity of glass K9 vs. temperature. (□) Without radiation, (○) with radiation of the sample alone, (△) with radiation of the wire and the sample; (—) fitting of experimental points.

As predicted by microscopic theoretical models of the thermal conductivity of disordered systems in solid-state physics, the temperature dependence of λ is similar to that of its specific heat. As far as glass is concerned, the variation of its thermal conductivity with temperature should be approximately linear, even at high temperatures. The thermal conductivity tends not to increase steeply, but rather to increase gradually, and this prediction has been confirmed by experimental results [7].

6. CONCLUSION

Based on the numerical analysis of the combined transient conductive–radiative heat transfer inside a semitransparent sample for the case of the hot-wire technique, a new simple analytic model for the thermal conductivity measurement of semitransparent solid materials has been developed. Two important factors, i.e., the overall effect of emission and absorption inside the sample and the overall effect of emission and absorption by the wire surface, are taken into account simultaneously in the new, simplified model. Based on parameter estimation, sensitivity analyses were performed for the new model, and it was found that three unknown parameters, the thermal conductivity of the sample, the thermal contact resistance, and the effective mean absorption coefficient, are uncorrelated and can be determined simultaneously. Furthermore, an experimental study was carried out on one special glass—glass K9—in the temperature range of 297 to 1230 K, and the radiation-free thermal conductivity was determined. Our experimental results agree closely with the predictions of microscopic theoretical models of the thermal conductivity of disordered systems, and this result supports the applicability of the developed measurement technique.

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