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Thermal Diffusivity Measurements on Insulation Materials with the Laser Flash Method[1]

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An iterative approach is adopted to determine the thermal diffusivity of the xonotlite-type calcium silicate insulation material with very low thermal conductivity. The measurements were performed with a conventional laser flash apparatus by rear-face detection of the temperature response of the three-layered sample, where the insulating material is sandwiched between two iron slices. In the evaluation of the thermal conductivity, the theoretical curve is fitted to the complete temperature–time curve, instead of just using the $t_{1/2}$ point. The theoretical model is based on the thermal quadrupole method. The nonlinear parameter estimation technique is used to estimate simultaneously the thermal diffusivity, heat transfer coefficient, and absorbed energy. Based on experimental results, the optimal thickness range of the insulation material in the sample is indicated as 1.6 to 1.9 mm. The effects of the uncertainties of the thicknesses, contact resistance, and thermophysical properties of the three layers on the measurement uncertainty are estimated, giving an overall uncertainty in the thermal conductivity of approximately 7.5%.

KEY WORDS: insulation; laser flash; parameter estimation; thermal diffusivity; thermal quadrupole method; xonotlite-type calcium silicate.

1. INTRODUCTION

Thermal conductivities and thermal diffusivities of insulation materials are usually determined by steady-state or transient methods, such as the guarded hot-plate method, the radial heat-flow method, the hot-wire method, the

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hot-plane source method, etc. [1, 2]. The laser-flash method developed by Parker and his co-workers [3] has made rapid improvement in recent years, which has become one of the favored and accepted transient methods to determine the thermal diffusivity of materials such as metals, conductors, and semiconductors, as well as building materials, based on its simplicity of manipulation, accuracy of measurement, wide temperature range, and small sample size. This method has also been applied to thermal diffusivity measurements of layered structures [4, 5]. However, the laser flash method is seldom used to determine the thermal diffusivity of insulation materials because the heat loss of the sample cannot be ignored for a long heat transfer time, and there usually exists radiation penetration problems if the insulation sample is excited directly by the laser pulse.

In this paper we describe measurements of the thermal diffusivity of insulation materials by the laser flash method. The insulation material, xonotlite-type calcium silicate, is sandwiched between two iron slices to avoid radiation penetration. A nonlinear parameter estimation technique is adopted to estimate the thermal diffusivity of xonotlite. The theoretical curve is fitted to the complete temperature-time curve of the rear face of the sample, instead of just using the $t_{1/2}$ point. The theoretical temperature response model is based on the thermal quadrupole method [6]. Using a nonlinear parameter estimation algorithm, the thermal diffusivity of the insulation material and the heat transfer coefficient as well as the absorbed energy can be estimated simultaneously.

2. THEORETICAL MODEL

The physical model for the thermal diffusivity measurement is shown in Fig.1. The heat loss on the side face is neglected. If the sample is excited by a uniform laser pulse on the front surface, the one-dimensional heat transfer problem can be expressed by the following thermal quadrupole method [6]:

$$\begin{bmatrix} \theta_1 \\ \phi_1 \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_2 \\ \phi_2 \end{bmatrix} \tag{1a}$$

Here,

$$\begin{bmatrix} A & B \\ C & D \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ hS & 1 \end{bmatrix} \begin{bmatrix} A_1 & B_1 \\ C_1 & D_1 \end{bmatrix} \begin{bmatrix} 1 & R_t \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A_2 & B_2 \\ C_2 & D_2 \end{bmatrix} \begin{bmatrix} 1 & R_t \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A_1 & B_1 \\ C_1 & D_1 \end{bmatrix} \begin{bmatrix} 1 & 0 \\ hS & 1 \end{bmatrix} \tag{1b}$$

where R_t is the contact resistance, h is the heat transfer coefficient on both the front face and rear face, θ_1, ϕ_1 are the Laplace temperature and heat flow on the front face, respectively, θ_2, ϕ_2 are the Laplace temperature and

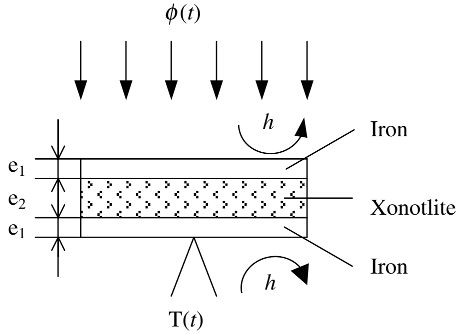

Fig. 1. Schematic of the physical model for thermal diffusivity measurements on insulation materials with the laser-flash method.

heat flow on the rear face, respectively, and S is the surface area. The coefficients in the matrix are as follows:

$$A_j = D_j = \cosh(e_j\sqrt{p/a_j}) \tag{2a}$$

$$B_j = \frac{1}{\lambda_j S\sqrt{p/a_j}} \sinh(e_j\sqrt{p/a_j}) \tag{2b}$$

$$C_j = \lambda_j S\sqrt{p/a_j} \sinh(e_j\sqrt{p/a_j}) \tag{2c}$$

where $a_j = \lambda_j/(\rho c_j)$, $j = 1$ or 2, p is the Laplace variable, a is the thermal diffusivity, λ is the thermal conductivity, ρ is the density, c is the specific heat, and e is the thickness of the sample.

With $\phi_1 = QS$ (the absorbed energy by the front face of the sample) and $\phi_2 = 0$ (no energy absorbed on the rear face), the temperature rise on the rear face is

$$\theta_2 = \frac{QS}{C} \tag{3}$$

with

$$\begin{aligned} C = {} & 2A_1A_2C_1 + A_1^2C_2 + B_2C_1^2 \\ & +2hS(A_1^2A_2 + A_1B_1C_2 + A_2B_1C_1 + A_1B_2C_1) \\ & +2hSR_t(A_1^2C_2 + 2A_1A_2C_1 + B_1C_1C_2) \\ & +2R_t(A_2C_1^2 + A_1C_1C_2) + 2(hS)^2R_t(A_1B_1C_2 + A_1^2A_2) \\ & +(hS)^2(2A_1A_2B_1 + A_1^2B_2 + B_1^2C_2) \\ & +R_t^2(C_1^2C_2 + 2hSA_1C_1C_2 + (hS)^2A_1^2C_2) \end{aligned} \tag{4}$$

Neglecting the contact resistance R_t, we have

$$C = 2A_1A_2C_1 + A_1^2C_2 + B_2C_1^2 + 2hS(A_1^2A_2 + A_1B_1C_2 + A_2B_1C_1 + A_1B_2C_1) + (hS)^2(2A_1A_2B_1 + A_1^2B_2 + B_1^2C_2) \quad (5)$$

If $F(p)$ is the known Laplace transform function of the rear-face temperature, the theoretical temperature rise on the rear face can be calculated by the Stehfest algorithm [7],

$$\theta_2^* = \frac{\ln(2)}{t} \sum_{i=1}^{n} v_i F(i \ln(2)/t) \quad (6)$$

3. PARAMETER ESTIMATION

A nonlinear parameter estimation process is needed to determine the thermal diffusivity of insulators. To complete this process, the following parameters should be known: the density, specific heat, thermal diffusivity, and thickness of the iron slice as well as the density, specific heat, and thickness of insulator. The specific heat values of iron and the insulator are measured with a differential scanning calorimeter (DSC) and the thermal diffusivity of iron is measured by the laser flash method. Some of these parameters are shown in Table I. Now there are still four unknown parameters: thermal diffusivity of insulator, thermal contact resistance, heat transfer coefficient, as well as the laser pulse energy absorbed by the sample. These four parameters have to be determined by the parameter estimation process. Here we analyze whether all of the four parameters can be estimated simultaneously. The sensitivity coefficients of the four parameters are shown in Fig. 2. The sensitivity coefficient is the first derivative of temperature with respect to the estimated property. In general, the sensitivity coefficients are desired to be large and uncorrelated (linearly independent). It is observed in Fig. 2 that the thermal diffusivity and thermal contact resistance are obviously correlated, so they cannot be estimated simultaneously. The thermal contact resistance is neglected in our study, and its effect on measurement precision will be discussed in the next section. The following three parameters have been adopted in the parameter estimation process instead of the direct estimation of a_2, h, and $Q: \beta_1 = a_2/e_2^2, \beta_2 = he_1/\lambda_1$, and $\beta_3 = \dfrac{Q}{2\rho_1 c_{p1} e_1 + \rho_2 c_{p2} e_2}$.

In the parameter estimation process, the initial β_1, β_2, and β_3 are given, and the iteration is carried out until the corresponding convergence condition is met. Here the residuals of the parameters are less than 0.1%. Improved Gaussian arithmetic has been adopted to complete the estimation process [8].

Table I. Specific Heat and Thermal Diffusivity of Iron and Specific Heat of Xonotlite

Temperature	(°C)	27	80	160	240	320	400
Iron slice	c_p $(\mathrm{J\cdot kg^{-1}\cdot K^{-1}})$	473	477.7	485.7	493.7	501.7	509.7
	a $(10^{-6}\mathrm{m^2\cdot s^{-1}})$	11.9	11.37	10.56	9.75	8.95	8.14
Xonotlite	c_p $(\mathrm{J\cdot kg^{-1}\cdot K^{-1}})$	823	912.6	988.5	1029.4	1052.7	1066.4

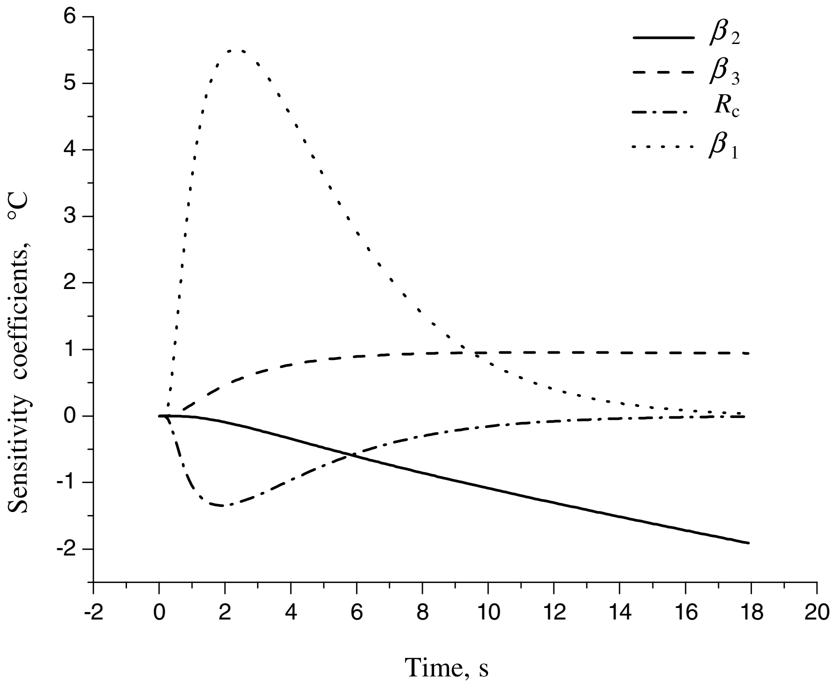

Fig. 2. Sensitivity coefficients for estimated parameters.

4. EXPERIMENTAL APPARATUS AND MEASUREMENT METHOD

The measurements are conducted on a LTC-1200 laser flash apparatus. A Nd Glass laser (1.06 μm) produces a beam of intensity 10–30 J with a duration of less than 0.5 ms. The rear-face temperature of the sample is detected through the thermocouple welded on the rear face of the sample. The signal from the thermocouple is amplified by a fast preamplifier. A DL-708E type digital oscilloscope then digitizes the analog signal before being processed by a computer.

The front surface of the three-layered sample has been sprayed with a black paint with high emissivity in order for the sample to absorb enough energy. Gold has been coated on the opposite surfaces of the two iron slices (both of the thicknesses are 0.45 mm) to increase the reflectivity and decrease direct radiation heat transfer between the two inner surfaces, because the insulation material has very high porosity.

5. RESULTS AND DISCUSSION

5.1. Experimental Results

Figure 3 shows a typical plot of the experimental temperature response and the calculated theoretical curve. From the figure we show that they coincide and the temperature response time is longer due to the excellent insulation capability and large specific heat of insulation materials. Since the heat loss of the sample is determined by estimating the heat transfer coefficient simultaneously, the thermal diffusivity measurement of the insulation material can be completed. We selected 1–15 s as the time region for parameter estimation, considering that the initial disturbance of the system may affect the estimation precision and the sensitivity coefficient of β_1 is small in the final stage. At room temperature and ambient pressure, we measured the

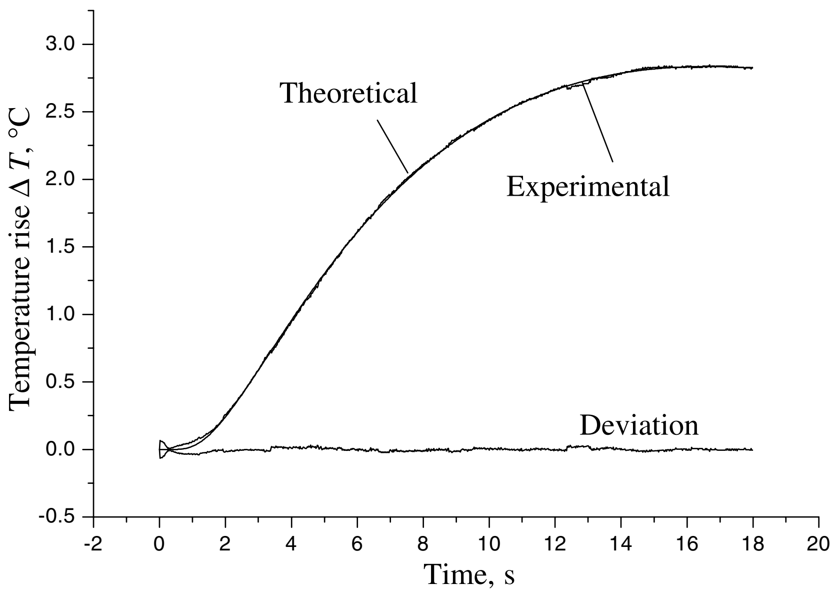

Fig. 3. Temperature response curve in parameter estimation.

thermal diffusivity of xonotlite-type calcium silicate with $\rho = 220\,\text{kg}\cdot\text{m}^{-3}$. The thermal conductivity of the same sample measured in this work by the hot-wire method is 0.063 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ with an experimental uncertainty of approximately 3%.

Figure 4 shows the relation of the thermal conductivity of xonotlite with the sample thickness. We notice that the thermal conductivity of xonotlite is essentially a constant for sample thicknesses larger than 1.3 mm. The results are very close to the value that is measured by the hot-wire method. It indicates that our xonotlite is an optically thick media when its thickness is larger than 1.3 mm, and the measured thermal conductivity value has contributions from both conduction and thermal radiation. To attain the expected measurement accuracy, we suggest a sample thickness of xonotlite of 1.6–1.9 mm, and this can satisfy both the optical thickness condition and a sufficient rear-face temperature response signal. The maximum temperature rise of the rear face of the sample is between 1.5 and 2.5 °C. A set of measurement results is shown in Table II.

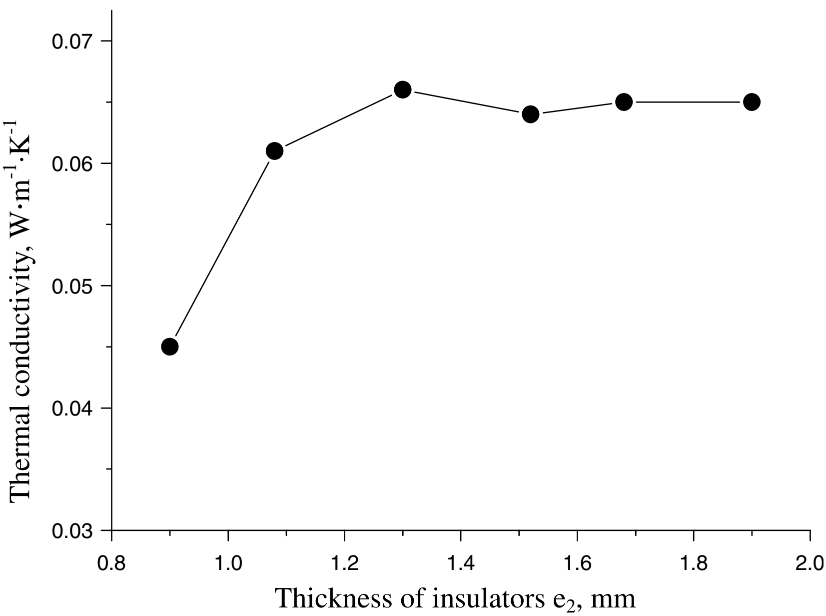

Fig. 4. Thermal conductivities of xonotlite measured with the laser flash method.

Table II. Measured Thermophysical Properties Data of Xonotlite with $\rho = 220\,\mathrm{kg \cdot m^{-3}}$

Times	1	2	3	4	5	6	Mean value
a $(10^{-6}\mathrm{m^2 \cdot s^{-1}})$	0.362	0.350	0.360	0.357	0.343	0.351	0.354
λ $(\mathrm{W \cdot m^{-1} \cdot K^{-1}})$	0.0655	0.0633	0.0652	0.0647	0.0621	0.0635	0.0641

Table III. Uncertainty Analysis in the Parameter Estimation Process

	e_1	e_2	R_t	$\rho_1 c_{p1}$	$\rho_2 c_{p2}$	a_1	Total
Maximum uncertainty	0.01×10^{-3} m	0.02×10^{-3} m	$2.45°\mathrm{C \cdot W^{-1}}$	3%	3%	3%	
Uncertainty of thermal conductivity	0.52%	3%	4.9%	3.1%	2.2%	2.8%	7.5%

5.2. Uncertainty Analysis

The overall measurement uncertainty comes mainly from the uncertainties in the sample thickness, the thermal contact resistance, the thermophysical properties of the three layers, and the inhomogeneity of the laser pulse as well as the asymmetry of the heat transfer coefficient. The influence of e_1 (thickness of iron slice), e_2 (thickness of insulator), and R_t as well as the thermophysical properties of the three layers on the parameter estimation result is summarized here, as shown in Table III.

6. CONCLUDING REMARKS

This paper presents a study of thermal diffusivity measurements on an insulation material with the laser flash method. Using the parameter estimation process and the sandwiched structure, this technique is convenient to measure the thermal diffusivity of insulators. This method can be used to measure thermal diffusivities of other similar insulation materials. A typical advantage of this method is that a very small sample is needed with a very rapid measurement process.

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