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# Rapid thermal conductivity measurement of porous thermal insulation material by laser flash method

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## ABSTRACT

Measuring the thermal conductivity of a high performance thermal insulation material within a short time is challenging. In the present study, a laser flash method is adopted to measure the thermal diffusivity of porous thermal insulation material. Small sample size and short measuring time are major advantages of this method. The specific heat capacity was measured by a differential scanning calorimeter (DSC). With known density, the thermal conductivity was obtained. Applying a calibration routine, the measured thermal conductivity of the porous material is only 0.039 W/(m K) at room temperature. This value is in good agreement with the data obtained using conventional method. It demonstrates that the laser flash method is a reliable technique to determine the performance of a very low thermal conductivity ity material.

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### 1. Introduction

The issues of global warming and energy shortage are becoming serious recently. People have tried to develop various kinds of thermal insulation materials to improve heat pollution. High performance thermal insulation materials are usually porous and exhibit very low thermal conductivity. The determination of the thermal properties for such material is the key issue for their development. However, to measure the materials with low thermal conductivity is very challenging.

There are two ways to measure the thermal conductivity: steady-state and transient methods [1–5]. Steady-state method gives a thermal gradient to test specimen, and measures the thermal conductivity directly. Steady-state method is frequently used to measure the thermal conductivity of thermal insulation material. However, this method needs relatively large specimen and long measuring times [1,2]. On the other hand, transient method provides transitional thermal flow to the specimen, then determines the thermal conductive properties from the response of the temperature change [3–5]. The heating modes, such as pulse

heating method, periodic heating method, and step heating method are frequently used. Laser flash heating mode is widely used. The technique uses small specimen; the measuring time is as short as a few minutes [3]. However, the transient methods determine the thermal diffusivity only; the specific heat capacity and density data are needed before calculating the thermal conductivity.

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In the present study, the thermal diffusivity of a porous thermal insulation material is measured with the laser flash method. Fig. 1 shows the principle and the theoretical temperature rise as a result from the laser flash heating. As a laser beam is spotted onto a solid disc in vacuum, a pulse heat is applied. At the same time, the temperature change at the back of the specimen was monitored. The temperature rise with time corresponds to the thermal energy diffusion along the thickness direction in the specimen. Therefore, the thermal diffusivity along the thickness for specimen disc can be obtained [3]. The thermal diffusivity  $\alpha$  is calculated from the following equation:

$$\alpha(T) = 0.1388 \times \frac{d^2}{t_{1/2}} \tag{1}$$

where *T* is the measuring temperature; *d* is the thickness of specimen;  $t_{1/2}$  is the time needed for the back of the specimen to reach

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Fig. 1. The principle and temperature rise curve of laser flash method.

half the maximum temperature after pulse heating, as shown in Fig. 1.

In order to obtain the thermal conductivity, specific heat capacity of specimen was measured using a differential scanning calorimeter (DSC). It is a reliable method to determine specific heat capacity. This method exhibits the features of using small specimen as well [6].

So far in this research area, Wei et al. measured the xonotlitetype calcium silicate thermal insulation material by a conventional laser flash apparatus. The thermal conductivity of xonotlite around 0.064 W/(m K) was measured in their report [7]. However, in this study, the advanced insulation material with much lower thermal conductivity was successfully developed and measured by laser flash method.

In this paper, the thermal conductivity for porous thermal insulation material at room temperature is measured and compared with the data obtained by conventional methods. As a result, it suggests the proposed method is very promising for rapid measurement of very low thermal conductivity materials.

#### 2. Experimental procedures

The details for the preparation of the thermal insulation material with nano porous structure can be found in a previous report [8]. A brief on the preparation procedures is given here. The starting materials were fumed silica powder (AEROSIL300, Nippon Aerosil, Tokyo, Japan), glass fiber (CS3J-888, Nittobo, Tokyo, Japan) and SiC powder (Silcar-G1, Wacker Chemie, Munich, Germany). The specific surface area of the fumed silica was 300 m<sup>2</sup>/g as measured by BET method. The size of fumed silica was 10 nm as estimated from specific surface area. The fumed silica powder showed a three-dimensional nano chain-like structure with the size of several tens nanometers [9,10]. The length and diameter of the glass fiber were around 3 mm and 10 um, respectively. The average diameter of SiC particle was 3.3 µm.

The ratio (mass%) of fumed silica:glass fibers:SiC in the powder mixture was 60:20:20. A mechanical processing was applied for the starting powder mixture by an attrition type mill (Mechanofusion System, Hosokawa Micron Corp., Osaka, Japan). The processing time was only 10 min. The mill device composed of main chamber, and certain clearance between the rotating rotor and



Fig. 2. A typical temperature rise curve for the specimen #1.



Fig. 3. Apparent thermal diffusivity as a function of amplitude of output signal.

Table 1

Thermal diffusivity measured by laser flash method at room temperature (300 K).

	Average thickness (mm)	Standard deviation of thickness (mm)	Thermal diffusivity (m²/s)
Specimen #1 Specimen #2 Specimen #3 Specimen #4	2.304 2.522 2.331 2.564	0.050 0.065 0.066 0.039	$\begin{array}{c} 1.2\times 10^{-7}\\ 1.4\times 10^{-7}\\ 1.2\times 10^{-7}\\ 1.4\times 10^{-7}\end{array}$

chamber [11]. The applied rotating speed and clearance of the device were 1000 rpm and 3 mm, respectively. After the mechanical processing, the obtained composite powder was uniaxially pressed to form the bulk compact (100 mm  $\times$  150 mm  $\times$  10 mm) with pressure of 2 MPa at room temperature. During the processing, the temperature and humidity were approximately 25 °C and 50%, respectively. By measuring the compact's dimensions and weight, its apparent density and porosity were determined.

The specimens for the measurement of thermal diffusivity and heat capacity were cut from the above porous compact. The thermal diffusivity was measured by laser flash apparatus (LFA-502N, Kyoto Electronics Manufacturing CO., LTD, Japan) at room temperature (near 300 K) in air. The size of the specimens was 10 mm in diameter and 2.5 mm in thickness. As the nano porous compact is very fragile, the cutting process should be very cautious. First we cut the thin board with 2.5 mm thickness from the compact carefully by sawing machine. Then, the belt punch with the diameter

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