Measurement of Thermophysical Properties of Fire Testing Materials*

Masatoshi DAIKOKU** and Kozo SAITO***

An unsteady heat flux method was used to determine the thermophysical properties of Polymethyl methacrylate (PMMA) and cellulose, both of which are commonly used as standard sample materials in flame spread tests. The analysis is based on an analytical solution to a heat conduction equation with periodic heat flux. The effective thermal conductivity and diffusivity for a packed bed of each sample material were simultaneously determined from the amplitude and phase delay of the temperature response for periodic heat flux. Experiments were performed for a range of heating rates, particle sizes and apparent densities. The variation in thermophysical properties with temperature was obtained, and the effects of pyrolysis for cellulose, and phase transition for PMMA, on the thermophysical properties of these materials were investigated.

Key Words: Thermophysical Property, Packed Bed, Heat Conduction, Thermal Conductivity, Thermal Diffusivity, Cellulose, PMMA, Fire

1. Introduction

Studies on the flame spread characteristics of common building materials are important in fire safety engineering. In our previous research on room fire, we focused on upward flame spread along the corner between two walls, and an extensive study has been carried out using a half-scale room corner model. This research began with the observation of the flow of fire in rooms⁽¹⁾, and is now nearing completion with the performance of flame spread tests^{(2),(3)} for standard materials such as Polymethyl methacrylate (PMMA) and Douglas Fir Particle Board (DFPB).

The results of the prior research have revealed that the highest rate of flame spread on walls does not occur at the vertical corner; the highest rates actually occur a few centimeters from the corner. It was also found that the upward flame spread rate was extremely high on preheated walls. Although the

surface temperature and incident heat flux profiles for the wall corners have been measured for noncombustible materials such as Marinite Board⁽⁴⁾, such analysis have not been conducted for combustible materials where pyrolysis may take place. Therefore, gaining an understanding of the pyrolysis of building materials is important and necessary from a fire safety point of view.

A highly accurate model has been developed for well-defined ideal materials in simple geometries^{(5),(6)}, however experiments still play a major role in investigating pyrolysis phenomena. In our previous study⁽⁷⁾, the possible use of cellulose as a benchmark sample for pyrolysis and flammability tests was discussed, and the use of effective thermal conductivity for charring materials such as DFPB and the in-depth radiation absorption effects in PMMA were evaluated. The effective thermal conductivities of DFPB and pure cellulose were approximated by fitting the measured temperature profiles to the formula defined as $\lambda_{eff} = \lambda_s(T) + \lambda_r(T^3)$, where $\lambda_s(T)$ is the thermal conductivity of the solid and $\lambda_r(T^3)$ is the effective radiation contribution.

In the development of theoretical models that incorporate both pyrolysis and flame spread, temperature-dependent thermophysical properties and the porosity of the sample must be accounted for to

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** Department of Mechanical Engineering, Hachinohe Institute of Technology, 88-1 Myo Ohbiraki, Hachinohe 031-8501, Japan. E-mail: daikoku@hi-tech.ac.jp

*** Department of Mechanical Engineering, University of Kentucky, 514 B CRMS Building, Lexington, Kentucky 40508-0108, USA

accurately describe heat transfer in the materials. However, thermophysical properties data for standard materials has not been compiled for a wide range of temperatures, in this case to ignition temperature. The objective of this study is to obtain reliable thermophysical properties data (thermal conductivity and thermal diffusivity) for standard materials. Pure cellulose is used as a charring material, and PMMA as a non-charring material.

In this paper we discuss the validity of measuring thermophysical properties using an unsteady heat flux method, examine the variations in effective thermal conductivity and diffusivity with temperature for packed beds of PMMA and pure cellulose, and investigate the effect of pyrolysis and phase change of the samples on the thermophysical properties of the materials.

Nomenclature

- a : line heater radius
 b : cylinder radius
 d_p : particle diameter
 e : porosity of packed bed
 Fo : Fourier number $(=\kappa t_p/a^2)$
 h_{rs}, h_{rv} : heat transfer coefficient in Kunii-Smith's equation (Eqs.(18), (19) and (20))
 q_0 : amplitude of sinusoidal heat flux from line heat source
 r : radial distance
 R : nondimensional radial distance $(=r/a)$
 R_b : nondimensional cylinder radius $(=b/a)$
 t : time
 t_p : period of periodic temperature component
 T : temperature in Kelvin (Eqs.(19), (20))
- Greek letters
- ε : emissivity in Kunii-Smith's equation (Eqs.(19), (20))
 η : amplitude of periodic temperature component
 $\bar{\eta}$: nondimensional amplitude of periodic temperature component $(=\lambda\eta/q_0a)$
 θ : temperature
 κ : thermal diffusivity
 λ : thermal conductivity
 ξ : nondimensional constant $(=(2\pi/Fo)^{1/2})$
 ρ_{app} : apparent density of packed bed
 τ : nondimensional time $(=\kappa t/a^2)$
 ϕ : phase delay between line heat source and arbitrary radius

2. Measurement Principle

Figure 1 shows an illustration of the cylindrical cell that will be used to explain the measurement principle⁽⁸⁾. The line heat source supplies a sinusoidal

Fig. 1 Measurement principle

heat flux from its surface ($r=a$) into the sample in the cylinder.

In the theoretical analysis for determining thermophysical properties, the following assumptions are adopted:

1) The sample is packed into a cylindrical cell, and the radius and length of the cylinder are regarded as infinite. The heat flow is one dimensional (radial).

2) The thermophysical properties of the sample are considered to be constant for the period of measurement t_p .

3) The elapsed time from the beginning of the sinusoidal heating process is very long.

4) The periodic component can be extracted accurately from the measured temperature. The ramped component due to continuous heating is also included in the measured temperature. The validity of this assumption was discussed in the previous study⁽⁹⁾.

The heat conduction equation with initial and boundary conditions based on assumptions 1) and 2) is given by

$$\frac{\partial \theta}{\partial t} = \kappa \left(\frac{\partial^2 \theta}{\partial r^2} + \frac{1}{r} \frac{\partial \theta}{\partial r} \right) \quad (1)$$

Initial condition:

$$\theta = 0 \quad \text{at} \quad t = 0 \quad (2)$$

Boundary conditions:

$$-\lambda \frac{\partial \theta}{\partial r} = q_0 \cos \frac{2\pi t}{t_p} \quad \text{at} \quad r = a \quad (3)$$

$$\theta = 0 \quad \text{at} \quad r = b \quad (4)$$

Under assumption 3), the solution of the heat conduction equation (1) at an arbitrary radius r can be given by the following equation:

$$\theta = \eta \cos \left(\frac{2\pi \tau}{Fo} - \phi \right) \quad (5)$$

where η is the amplitude of the periodic temperature component under assumption 4), and ϕ is the phase delay between the line heat source and the arbitrary radius. Nondimensional time τ and Fourier number Fo are defined as

$$\tau = \frac{\kappa t}{a^2} \quad (6)$$

$$Fo = \frac{\kappa t_p}{a^2} \quad (7)$$

The amplitude in Eq. (5) is written in the form:

$$\eta = \frac{q_0 a}{\lambda} \bar{\eta} \quad (8)$$

where $\bar{\eta}$ is the nondimensional amplitude, given by

$$\bar{\eta} = \{ [Aber(\xi R) + Bbei(\xi R) + Cker(\xi R) + Dkei(\xi R)]^2 + [-Abei(\xi R) + Bber(\xi R) - Cker(\xi R) + Dkei(\xi R)]^2 \}^{0.5} \quad (9)$$

Here, *ber*, *bei*, *ker*, and *kei* are Kelvin functions, and $R (= r/a)$ is a nondimensional radius. The phase delay ϕ in Eq. (5) is given in the form

$$\phi = \tan^{-1} \frac{-Abei(\xi R) + Bber(\xi R) - Ckei(\xi R) + Dker(\xi R)}{Aber(\xi R) + Bbei(\xi R) + Cker(\xi R) + Dkei(\xi R)} \quad (10)$$

where

$$\begin{bmatrix} ber'(\xi) & bei'(\xi) & ker'(\xi) & kei'(\xi) \\ -bei'(\xi) & ber'(\xi) & -kei'(\xi) & ker'(\xi) \\ ber(\xi R_b) & bei(\xi R_b) & ker(\xi R_b) & kei(\xi R_b) \\ -bei(\xi R_b) & ber(\xi R_b) & -kei(\xi R_b) & ker(\xi R_b) \end{bmatrix} \times \begin{bmatrix} A \\ B \\ C \\ D \end{bmatrix} = \begin{bmatrix} -\xi^{-1} \\ 0 \\ 0 \\ 0 \end{bmatrix} \quad (11)$$

$$\xi = \sqrt{\frac{2\pi}{Fo}} \quad (12)$$

$$R_b = \frac{b}{a} \quad (13)$$

As mentioned in assumption 1), the radius of the cylinder b is so large compared to the distance between the centerline and the thermocouple junction at r that the following approximations can be made:

$$ker(\xi R_b) = 0 \quad (14)$$

$$kei(\xi R_b) = 0 \quad (15)$$

Therefore, the nondimensional amplitude $\bar{\eta}$ and the phase delay ϕ in Eqs. (9) and (10) can be simplified as

$$\bar{\eta} = \sqrt{\frac{ker^2(\xi R) + kei^2(\xi R)}{\frac{2\pi}{Fo} \{ker'^2(\xi) + kei'^2(\xi)\}}} \quad (16)$$

$$\phi = \tan^{-1} \frac{kei'(\xi)ker(\xi R) - ker'(\xi)kei(\xi R)}{ker'(\xi)ker(\xi R) + kei'(\xi)kei(\xi R)} \quad (17)$$

Equation (17) implies that the phase delay is a function of the Fourier number, which is itself a function of thermal diffusivity κ . Furthermore, the amplitude in Eq. (8) contains thermal conductivity λ and the nondimensional amplitude, which is a function of the Fourier number. Hence, the thermal conductivity and diffusivity are obtained from experimental values of the phase delay and amplitude.

3. Experimental Procedure

3.1 Experimental apparatus

Figure 2 shows a schematic of the test cell. Nichrome wire of 0.18 mm diameter was used as a line heat source, placed along the centerline of the cylindrical stainless cell (40 mm ID $\times$ 104 mm length). A Chromel-Alumel thermocouple of 0.1 mm diameter was placed parallel to the heat source, 5 mm from the center, and the position of the Chromel-Alumel junction was set at midway along the cylinder. The line heat source and the thermocouple were inserted into alumina sheaths of 0.5 mm OD $\times$ 0.2 mm ID and 0.8 mm OD $\times$ 0.4 mm ID, respectively. In the present study the inner radius of the alumina sheath was taken as the radius of the line heat source, i.e. $a = 0.2$ mm. In order to estimate the heat flux value q_0 , copper wires were attached to both ends of the heater to measure the voltage. To prevent displacement of the line source and the thermocouple, the sample was packed into the cylinder through the two holes in the end of the cylinder after attaching the two alumina sheaths.

Figure 3 shows a schematic of the experimental apparatus. The line heat source was connected to a DC power supply controlled by a personal computer via a GPIB programmer. The cylinder was housed in an electric furnace, allowing the temperature of the entire cell to be heated at a constant rate while varying the heat flux from of the line heater sinusoidally with time. The electromotive force of the thermocouple, and the heater voltage, were measured with a multi-logger connected to the computer.

To generate a sinusoidal heat flux, the voltage load on the heater was varied in proportion to $[1 + \cos(2\pi t/t_p)]^{1/2}$. Sinusoidal heat generation was

Fig. 2 Test cell

Fig. 3 Schematic of experimental apparatus

confirmed in a preliminary experiment.

3.2 Determination of thermophysical properties

After acquiring the temperature data, the thermal conductivity and diffusivity were calculated by the following procedure:

- 1) The periodic component was extracted from the measured temperature.
- 2) The amplitude and phase delay were determined from the periodic temperature component.
- 3) The Fourier number Fo was determined from the phase delay by Eq.(17). The thermal diffusivity was obtained from the definition of Fo in Eq.(7).
- 4) The nondimensional amplitude was determined from Fo by Eq.(16). The thermal conductivity was then obtained by Eq.(8).

3.3 Experimental conditions

A packed bed of glass particles, a non-reactive material for which there is ample thermophysical data, was used to check the accuracy of the present measurement technique. The mean particle sizes adopted were 0.05, 0.1, 0.4, and 1.0 mm. Table 1 shows the properties of cellulose and PMMA used in this study, along with their chemical components and the temperature range of decomposition⁽¹⁰⁾. For pure cellulose powder, two samples were prepared; grade # 40-100 and grade # 200+ (mesh size). The apparent density of the packed bed was varied from approximately 350 kg/m³ to approximately 500 kg/m³. For the PMMA sample, mono-dispersed fine particles of 10, 20, and 60 μ m in diameter were adopted, and the apparent density of the sample was varied from approximately 700 kg/m³ to approximately 800 kg/m³. The cylindrical sample was heated from 300 K (room temperature) to around 1100 K at constant heating rates of between 0.01 and 0.08 K/s. As large spatial temperature differences (radial) may cause measurement error, the amplitude of the periodic heat flux for each heating rate was determined in the preliminary experiment so as to keep the temperature profile as uniform as possible.

Table 1 Properties of samples

	Cellulose	PMMA
Structural formula	$\left[\begin{array}{c} \text{CH}_2\text{OH} \\ \\ \text{C} - \text{O} - \text{C} \\ \quad \\ \text{OH} \quad \text{OH} \end{array} \right]_n$ $(\text{C}_6\text{H}_{10}\text{O}_5)_n$	$\left[\begin{array}{c} \text{CH}_3 \\ \\ \text{C} - \text{CH}_2 \\ \\ \text{COOCH}_3 \end{array} \right]_n$ $(\text{C}_5\text{H}_8\text{O}_2)_n$
Decomposition temperature ⁽¹⁰⁾ [K]	553-653	453-553
Particle diameter	#40-100 (mesh size)	10 micron
	#200 and greater	20
		60
Apparent density [kg/m ³]	350-500	700-800

4. Results and Discussion

4.1 Experimental validation of measurement technique

Prior to measuring the thermophysical properties of materials with unknown thermophysical characteristics, the validity of the present measurement technique must be confirmed. The values of thermal conductivity and diffusivity for a packed bed of glass particles obtained in this experiment were compared with the values estimated by the Kunii-Smith equation^{(11),(12)}, which gives the effective thermal conductivity λ_{eff} for a packed bed (porosity e) of solid particles of uniform diameter d_p

$$\frac{\lambda_{eff}}{\lambda_F} = \left(1 + \frac{h_{rv} d_p}{\lambda_F} \right) e + \frac{1-e}{\frac{1}{\Phi} + \frac{h_{rs} d_p}{\lambda_F} + \frac{2}{3} \frac{\lambda_F}{\lambda_s}} \quad (18)$$

where λ_s and λ_F are the thermal conductivities of a solid particle and its surrounding fluid, respectively. The parameter Φ is affected by the geometric arrangement of solid particles in the packed bed layer, and varies according to the thermal conductivity ratio of solid to fluid (λ_s/λ_F). The radiative heat transfer coefficients for the solid particle (emissivity ϵ) h_{rs} and for the fluid h_{rv} in Eq.(18) are expressed by the following equations:

$$h_{rs} = 0.2268 \frac{\epsilon}{2-\epsilon} \left(\frac{T}{100} \right)^3 \quad (19)$$

$$h_{rv} = \frac{0.2268}{1 + \frac{e}{2(1-e)} \frac{1-\epsilon}{\epsilon}} \left(\frac{T}{100} \right)^3 \quad (20)$$

Figure 4 shows a representative example of the effective thermal diffusivity of the glass particle bed. The particle size and the apparent density are 0.05 mm and 1070 kg/m³, respectively. The dotted line is the effective thermal diffusivity obtained from the effective thermal conductivity in the Kunii-Smith equation (18) and the heat capacity (C_p) for each phase (solid and fluid), given by

Fig. 4 κ_{eff} vs. temperature for packed glass particles

(a) Effective thermal conductivity

(b) Effective thermal diffusivity

Fig. 5 Variation in thermophysical properties with temperature for pure cellulose (# 200 and greater)

$$\kappa_{eff} = \frac{\lambda_{eff}}{(C\rho)_s(1-e) + (C\rho)_{fe}} \quad (21)$$

The experimental values were found to be in good agreement with the calculated values, for all particle sizes (0.05 mm to 1.0 mm), demonstrating that this measurement technique is accurate.

4.2 Thermophysical properties of cellulose

Figures 5(a) and 5(b) show variations in the effective thermal conductivity and diffusivity, respectively, of pure cellulose powder of grade # 200 and greater for 4 apparent densities. It was found that the effective thermal conductivity and effective thermal diffusivity increased almost linearly with temperature

apart from a significant peak and dip between 500 K and 680 K corresponding to pyrolysis. There are a number of proposed models of cellulose pyrolysis, and the measured temperature range of pyrolysis differ between them due to differences in experimental conditions. For the pyrolysis of cellulose, two main mechanisms have been identified⁽¹³⁾: One mechanism, which affects cellulose at temperatures of 500 K or lower, involves a reduction in the degree of polymerization by bond scission, the elimination of water, and the formation of low molecular weight gases and char residue. Above temperatures of about 570 K the second mechanism becomes dominant, and involves two reactions; the production of char and gas, and the production of tar. This reaction of the second mechanism is complete by 650 K⁽¹⁴⁾. Our result suggests that the cellulose finishes pyrolysis at around 680 K, from the thermophysical properties data, and subsequently exists in a charred state. This is confirmed by the fact that the sample changed from white to dark brown and lost mass due to the evaporation of water and the release of volatile gases. The effective thermal conductivity and effective thermal diffusivity of the charred sample then continues to increase above 680 K, behaving similar to other charring materials such as coal⁽⁹⁾.

The pyrolysis of cellulose, which occurs at between 500 K and 680 K, agrees with the decomposition temperature range shown in Table 1. Pyrolysis, which is an endothermic reaction, is most active for cellulose at around 600 K, and therefore, both thermal conductivity and diffusivity have maximum values for the original state of the material at this temperature. The apparent density had little effect on the thermophysical properties, however this observation is complicated by the fact that the apparent density may change due to the mass loss during measurement. Furthermore, the continuous heating rate, except the 0.08 K/s case, was not observed to have any significant effect on the thermophysical properties of the material in the present experiment. This means that pyrolysis is dominant in the heating process, in other words, the pyrolysis rate was much higher than the continuous heating rate of the sample. However, it should be noted that no heat generation term is introduced in the heat conduction equation (1) of the measurement principle. If the radial temperature distribution were kept uniform from the center to the inner surface of the cylinder, however, the effect of exothermic or endothermic reactions on the thermophysical properties would be minimized⁽¹⁵⁾. At the heating rate of 0.08 K/s, the thermophysical properties data was somewhat scattered, as it is difficult in such a case to maintain a uniform temperature distri-

Fig. 6 Variation in thermophysical properties with temperature for PMMA (heating rate=0.05 K/s)

bution during measurement, measurement error increases with the heating rate.

4.3 Thermophysical properties of PMMA

Figures 6(a) and (b) show variations in the effective thermal conductivity and diffusivity of a fine PMMA particle bed. The continuous heating rate was found not to have an appreciable effect on the thermophysical properties of PMMA in the preliminary experiment, and a constant heating rate of 0.05 K/s is shown as a representative case. PMMA, a thermoplastic resin that is well known as an amorphous polymer, begins to undergo thermal deformation at 373–378 K, and has a glass transition region⁽¹⁶⁾ at 374–399 K. The thermal conductivity of bulk PMMA has been reported to increase with temperature up to the glass transition and then decreases⁽¹⁶⁾. In the present experiment, the effective thermal conductivity increases rapidly with temperature up to 450 K, which exceeds the glass transition temperature, and then remains constant at 0.2 W/(m·K) regardless of particle size.

It is interesting that this value almost coincides with that of the thermal conductivity of bulk PMMA (0.21 W/(m·K))⁽¹⁷⁾, suggesting that the PMMA sample is no longer in the fine particle state at this temperature, instead forming an agglomerated mass at around 450 K or higher, corresponding to the rubber

state of bulk PMMA. Applying Kunii-Smith equation (18) to the 20 μm case, the calculated value of effective thermal conductivity is approximately 0.1 W/(m·K) at 400 K, which is in good agreement with the experimental value.

As shown in Fig. 6(b), particle size had no significant effect on effective thermal diffusivity in the low temperature region, although the smaller diameters produced a larger thermal diffusivity in the range 450 K to 600 K. The smaller particles seem to be directly affected by the heating rate, and thus the glass transition in the 10 μm case occurred ahead of the other two cases (20 and 60 μm). In the transition, the samples changed to the rubber state and melted. During extended heating processes, the material melted and volatilized⁽¹⁸⁾ such that no residue other than a small amount of carbonized matter was observed inside the cylinder after measurement. Therefore, further studies are needed in order to accurately take into account heat generation and mass loss during heating process^{(19),(20)}.

It was found that from the present measurements that thermal conductivity data generally tends to be somewhat scattered compared to diffusivity data, as shown in Figs. 6(a) and (b). Thermal diffusivity may be readily estimated using Eqs. (10), (12) and (7) if the phase delay is experimentally determined. By contrast, precise estimation of both the heat flux and heat flux amplitude in Eq. (8) is needed in order to accurately calculate the thermal conductivity. Therefore, a more precise experimental technique and exact estimation of heat flux is required to satisfy assumption 4) and calculation procedure 1) more strictly.

5. Summary

An unsteady heat flux method was applied to the calculation of the thermophysical properties of PMMA and cellulose as standard samples for ignition and flame spread tests. The effective thermal conductivity and diffusivity for a packed bed of each sample were measured. In summary:

1) The validity of the present measurement technique was confirmed by comparing the experimental values of the thermal conductivity and diffusivity of a bed of glass particles with the estimated values obtained from the Kunii-Smith equation.

2) The effective thermophysical properties of pure cellulose and their temperature dependency were determined. The pyrolysis of cellulose occurred between 500 K and 680 K, and both the thermal conductivity and diffusivity of the material in its original state have maximum values at around 600 K. The effects of the continuous heating rate and the apparent

density were found to be little effect on the thermophysical properties.

3) The effective thermophysical properties of PMMA and their temperature dependency were determined. Particle size was not observed to have any significant effect on the effective thermal diffusivity in the low temperature region, although the smaller diameter cases produce a larger thermal diffusivity in the range 450 K to 600 K. Again, the effects of the continuous heating rate and the apparent density on the thermophysical properties were found to be minor.

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