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Improvement of specific heat measurement by the flash method

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Abstract

A new method to improve the accuracy of the flash method for the measurement of specific heat and thermal conductivity has been proposed. A temperature rise versus time curve was replotted with non-dimensional time scaled with the time elapsed to reach the maximum temperature rise. And instead of comparing the measured temperature rise of the test sample with that of the standard material at a certain predetermined time, say, half time ($t_{1/2}$) or maximum temperature ($\Delta T_{\max}$), the rescaled temperature rise curve was integrated between $0.5t_{1/2}$ and $1.5t_{1/2}$ to increase the reproducibility and accuracy of the specific heat measurement. In order to minimize the error of the measurement a circular disc was used to cut off the excess flash energy input to the sample surface. It was found that the proposed method produces the measurement accuracy of the specific heat within 1.7% in comparison with the standard data.

Keywords: Specific heat; Flash method; Thermal diffusivity; Half time

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1. Introduction

There are many limitations in the accurate measurement of specific heat by a conventional method, differential scanning calorimetry (DSC). Its accuracy depends on the flatness of sample surface. It requires sample thickness less than 1 mm. In addition, the sample to be measured should have similar shape and mass with those of the standard sample. Since there are three steps in the measurement procedure of the DSC method, the measurement procedure takes quite a long time to complete the measurement.

One of the most popular methods used for thermal diffusivity measurement is the flash method proposed by Parker et al. [1]. They eliminated the inherent problem of contact resistance of the classical methods. And the short measurement time in the flash method minimizes the measurement error due to the heat losses from the sample to the surroundings. It provides both excellent accuracy and reproducibility. In the flash method the front side of the sample surface is subjected to a very short burst of radiant thermal energy. The resulting temperature rise on the rear side of the sample surface is measured, and thermal diffusivity can be determined by comparing the measured temperature rise with the appropriate mathematical model. Some of these models are the analytical equations of Parker et al. [1], Cowan [2], Cape and Lehman [3], and Clark and Taylor [4]. In the present experiment the thermal diffusivity data were analyzed using the Cape and Lehman equation [3] at room temperature.

The flash method was used to measure specific heat in two different ways. Watanabe [5] compared the maximum temperature ($\Delta T_{\max}$) in the temperature rise curve between the standard sample and the test sample to determine the specific heat, whereas Shinzato and Baba [6] compared the temperature rise at the half time $t_{1/2}$ on the temperature rise versus time curve where $t_{1/2}$ is the time at which the temperature rise is half of the maximum temperature rise. The measurement error by these methods is usually larger than 10%.

In the present study, we propose to integrate the temperature rise curve between $0.5t_{1/2}$ and $1.5t_{1/2}$ to determine the specific heat. This integration method was found to produce more reliable and accurate measurement of the specific heat.

2. Theory of measurement

The specific heat can be obtained using the flash method by comparing the temperature rises of both the standard and

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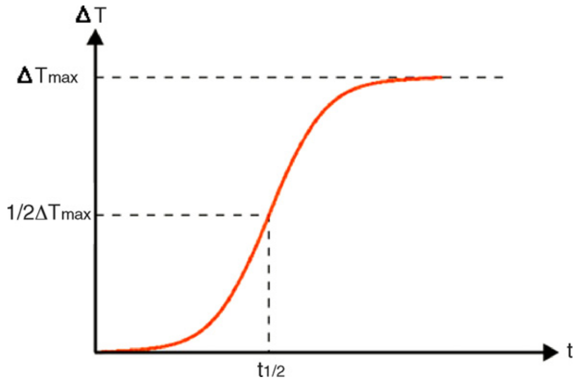

Fig. 1. Theoretical curve of temperature rise at the rear surface of the sample.

the test samples. If a sample is heated at the front surface, the temperature rise at the rear surface of the sample at any time (t) can be written as

$$\frac{\Delta T}{\Delta T_{\max}} = 1 + 2\left[\sum_{n=1}^{\infty}(-1)^n \exp(-n^2 2\pi^2 \alpha t \ell^{-2})\right] \qquad (1)$$

where α and ℓ are the thermal diffusivity and sample thickness, respectively, ΔT the temperature rise at the rear surface, $\Delta T_{\max}$ the maximum temperature rise at the rear surface, and t is time after pulse heating (Fig. 1).

Designating the time when the temperature rise reaches $\Delta T/\Delta T_{\max} = 1/2$ as $t_{1/2}$, the thermal diffusivity can be obtained from Eq. (1) as follows:

$$\alpha = \frac{0.138785\ell^2}{t_{1/2}} \qquad (2)$$

When the flash energy is irradiated uniformly at a rate Q per unit area and time and the absorptivity of both samples are same, the temperature rises ΔT_{s} and ΔT_{r} can be expressed by the following equations:

$$\Delta T_{\mathrm{s}} = \left(\frac{Q}{\rho \ell C_p}\right)_{\mathrm{s}} \qquad (3)$$

$$\Delta T_{\mathrm{r}} = \left(\frac{Q}{\rho \ell C_p}\right)_{\mathrm{r}} \qquad (4)$$

where the suffixes r and s are the standard and the test sample, respectively, and (is the sample density.

The specific heat of the test sample $C_{p\mathrm{s}}$ can be determined by comparing the temperature rise of the test sample to that of the standard sample with known specific heat under the same condition. Then the thermal conductivity (k) is obtained from $k = \rho\alpha C_p$.

Finally the test sample $C_{p\mathrm{s}}$ can be expressed as:

$$C_{p\mathrm{s}} = \frac{\rho_{\mathrm{r}} \ell_{\mathrm{r}} C_{p_{\mathrm{r}}} \Delta T_{\mathrm{r}}}{\rho_{\mathrm{s}} \ell_{\mathrm{s}} \Delta T_{\mathrm{s}}} \qquad (5)$$

This equation is made of the fact that the temperature rise is proportional to the output voltage (V) of the IR detector resulting from the absorbed flash energy divided by the detector amplifier gain (G).

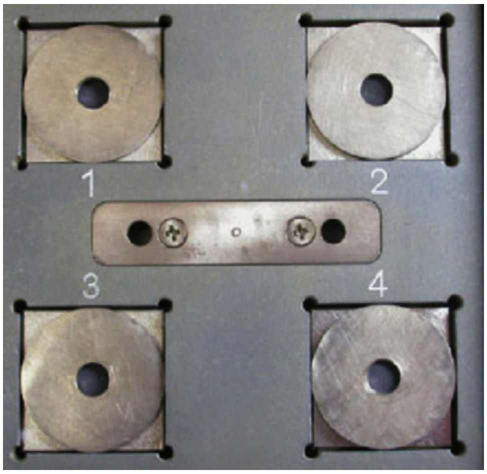

Fig. 2. Photographs of sample holder covered with circular discs having an inner hole.

3. Experimental procedure

The used standard samples were polycrystalline alumina, pyroceram 9606, pyrex 7790 supplied by Netzsch. Both sides of the samples were coated by spraying a thin layer of graphite. The purpose of coating is to increase the absorbance of flash energy on the front surface and to increase the intensity of the infrared light which is emitted from the rear surface. Fig. 2 shows the circular discs with an inner hole whose diameter is 6 mm. This circular disc is placed on top of the square sample of 8 mm in length to cut-off the excess flash beam and to allow a constant emission.

The experiments have been performed with a Xenon flash apparatus (Nanoflash LFA 447, Netzsch). A Xenon flash lamp is used to produce the heat pulse on the front surface of the sample. The length of the heat pulse can be varied from 0.1 to 0.5 ms. The samples are placed in an automatic sample changer which can test up to four samples in one test run. The measurement of the temperature rise on the rear of the sample surface is carried out with a liquid nitrogen cooled InSb (indium–antimonide) infrared detector.

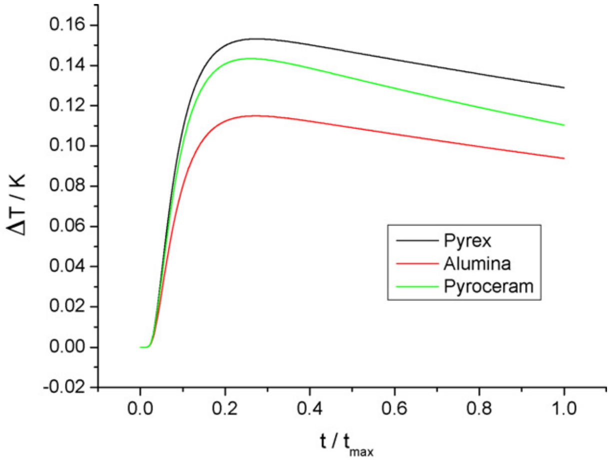

Fig. 3. The measured temperature rise curves for alumina ($G = 5002$), pyroceram ($G = 2520$) and pyrex ($G = 2520$).

Table 1
Comparison of measured thermal diffusivity (α_m) with standard one (α_s) for three sample materials

Material	ℓ (mm)	m (mg)	ρ (g/cm^3)	α_s (mm^2/s)	α_m (mm^2/s)	Error (%)
Alumina	0.998	0.2512	3.93	10.23	10.115	1.12
Pyrex	0.989	0.1375	2.22	0.650	0.646	0.62
Pyroceram	1.002	0.1677	2.62	1.926	1.923	0.16

Table 2
Comparison of the measured specific heats with standard values

Test	Standard	C_{pr} (J g^{-1} K^{-1})	C_{pm} (J g^{-1} K^{-1})	Error (%)
(a) Alumina	Pyrex	0.775	0.7798	−0.62
(b) Alumina	Pyroceram	0.775	0.7882	−1.71
(c) Pyrex	Pyroceram	0.761	0.7692	−1.08
(d) Pyrex	Alumina	0.761	0.7563	+0.61
(e) Pyroceram	Pirex	0.800	0.7941	+1.07
(f) Pyroceram	Alumina	0.800	0.7866	+1.68

4. Data analysis

In this paper a new method was proposed to determine the specific heat. In the laser flash method, the temperature change (ΔT) is usually measured for a time of t_{max} (defined as 15 times $t_{1/2}$) to determine the thermal diffusivity of samples. Since the decreasing rate of ΔT after the maximum temperature depends mainly on the material property, the value of t_{max} differs from sample to sample. If we scale the elapsed time with t_{max}, we can plot the ΔT data from different materials on the single graph with the axis of t/t_{max}. Then the temperature rise versus non-dimensional time t/t_{max} is integrated in the region between $0.5t_{1/2}$ and $1.5t_{1/2}$. This value is substituted for ΔT in Eq. (5) to determine the specific heat. Fig. 3 gives the plot of temperature rise versus non-dimensional time for three different materials.

The $0.5t_{1/2}$–$1.5t_{1/2}$ region was selected to compare the integrated values in the range from $0.5t_{1/2}$ to $1.5t_{1/2}$ on the temperature rise versus time curve for the three samples used. They were obtained by using the Trapezoidal rule as shown in Fig. 4.

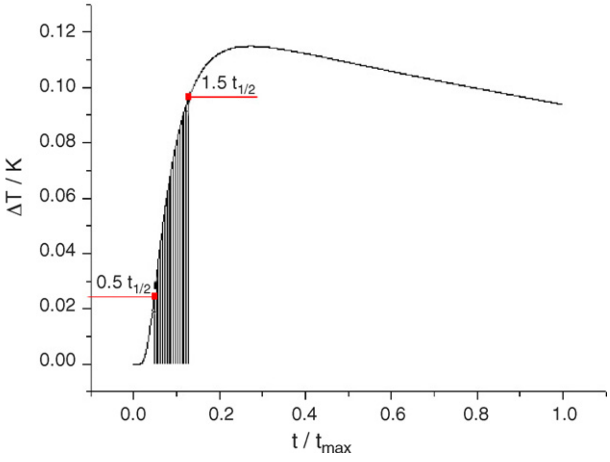

Fig. 4. Integration of the temperature rise curve from $0.5t_{1/2}$ to $1.5t_{1/2}$.

5. Results and discussions

Three types of materials (polycrystalline alumina, pyroceram 9606, and pyrex 7790) were tested using the circular disc having a constant inner hole. Table 1 presents the comparison of the thermal diffusivities of standard samples with the measured results by the flash method at room temperature.

The accuracy of specific heat were compared with six data from three materials which were calculated from Eq. (5).

Table 2 compares the measured specific heats (C_{pm}) with standard values (C_{pr}) for three test materials. It can be seen that the measured value depends on the selected material for the measurement. In all cases the error is within 1.7% at maximum.

We have used standard and test materials with marked difference in thermal diffusivity, while in general materials with similar thermal diffusivity were selected for the specific heat calculation for avoiding significant error associated with the method. In the new method measurement accuracy could be improved by introducing three innovative factors to the conventional flash method. They are: (1) use of circular disc to avoid input of excess flash energy, (2) selection of particular region of the time frame in the temperature rise versus time curve for determinination of the specific heat, and (3) introduction of non-dimensional time (t/t_{max}) instead of time (t) for the plot of temperature rise curve. With all of these factors, more accurate data could be obtained for materials having various thermal diffusivity values.

Another aspect of significant importance was selection of particular $t_{1/2}$ region for determinining specific heat measurement. Normally specifc heat was calculated using the region between $1t_{1/2}$ and $2t_{1/2}$ in the temperature rise versus t/t_{max} curve. However the region between $0.5t_{1/2}$ and $1.5t_{1/2}$ was found better to carry out the specific heat calculation.

6. Conclusions

In this study a new method has been proposed to improve the measurement accuracy of the current flash method to determine the specific heat and the thermal conductivity. A number of real measurements have been carried out to verify the accuracy and the reliability of the new flash method. It was found that the proposed method indeed improves the meaurement accuracy of the specific heat within 1.7% in comparison with standard data.

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