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THERMAL CONDUCTIVITY MEASUREMENTS USING THE FLASH METHOD

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Abstract

Thermal diffusivity is the speed with which heat propagates through a material. It has a multitude of direct applications, such as determining heat transfer through brake pads at the moment of contact, etc., but more often it is used to derive thermal conductivity from the fundamental relationship tying it with specific heat capacity and density.

Using a new multi-sample configuration system, and testing a reference sample adjacent to the unknown, specific heat capacity can be obtained parallel with thermal diffusivity. Thus, a single test yields thermal diffusivity and thermal conductivity with prior knowledge of density. The method is fast and produces results with high accuracy and very good repeatability. The sample size, 12 to 30 mm diameter and 2 to 5 mm thickness, is easy to handle and is well suited for a broad range of materials, even for composites, often a problem for other methods.

Typical data on two polymers, Pyrex glass and Pyroceram 9606 are presented.

Keywords: flash technique, specific heat capacity, Pyrex, Pyroceram, thermal conductivity, thermal diffusivity

General considerations

Thermal conductivity is one of the most sought after heat transport properties for any material. Measurement techniques for a long time have been based on models of uniaxial heat flow induced through the specimen, where thermal conductivity is obtained from the knowledge of the heat flux and the resultant thermal gradient along the sample. As a general rule, such model only operates in steady state conditions (which usually involve long settling times), and most importantly, accommodate no heat loss or gain to or from the surroundings. While this is easy to formulate on paper, it is exceedingly difficult to achieve in practice. There is a multitude of guarding schemes described in the literature, substantially diminishing in effectiveness and increasing in complexity with increasing operating temperatures. Generally, no effective guarding can be implemented above 1000°C, so the measurements' accuracy frequently falls well below 10%.

In more recent years, attention has been focused on thermal conductivity measurement techniques based on transient models. This was a natural evolution in measurement

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techniques, as advances in instrumentation caught up with theory. There are many non-steady state ways one can obtain thermal conductivity directly from the characteristics of heat propagation through a solid. Among these are the hot wire method, step-heat method, some surface probes or needle probes, differing mainly in the spatial geometry of heat propagation, the shape of the source (line or plate source), the nature of the heat pulse, and its frequency. Detailed discussion of these methods is beyond the scope of this paper, but it is sufficient to say that in many instances they work well, and are often more flexible than their steady-state counterparts. Because of their transient nature, they also can yield thermal diffusivity. The length of time the transient phenomenon takes place can vary from seconds to hours, depending on a multitude of equipment design parameters. Often, by selecting certain hardware and analysis of data, these tests can be performed very rapidly. In some cases, thermal diffusivity (or sometimes effusivity) is obtained, and thermal conductivity needs to be derived. For homogeneous materials, this can be done using a fundamental relationship

$$\lambda=\alpha\cdot\rho\cdot C_\mathrm{p} \tag{1}$$

where λ is thermal conductivity (W$\mathrm{m}^{-1}\mathrm{K}^{-1}$), α is thermal diffusivity ($\mathrm{m}^2\ \mathrm{s}^{-1}$), ρ is density (kg m^{-3}), and C_p is specific heat capacity (J $\mathrm{kg}^{-1}\mathrm{K}^{-1}$). It is obvious that prior knowledge of ρ and C_p is required in order to calculate thermal conductivity. Density is usually not a problem, as it can be determined from the sample's dimensions and its mass. Specific heat capacity is another matter. For totally homogeneous solids, one can resort to differential scanning calorimetry, but for coarse grain, layered, or otherwise heterogeneous materials, obtaining a minuscule sample that is representative for the material may be nearly impossible.

The flash method for measuring thermal diffusivity that has seen phenomenal growth and acceptance since its introduction in 1959 by Parker *et al.* [1], is one that has been limited by the same problem until the recent introduction of multiple sample instruments. The method, well described in the literature [2–7], is extremely elegant as it reduces a difficult thermal measurement to a much more manageable time measurement. It is based on depositing a very short but intense energy pulse on one surface of a disk shaped sample, while monitoring the temperature excursion of the opposite face. From the characteristic time dependence of the temperature rise, called thermogram, thermal diffusivity can be calculated using Parker's formula

$$\alpha=\frac{1.38d^2}{t_{1/2}} \tag{2}$$

where d is the sample's thickness and $t_{1/2}$ is the time necessary for the signal to reach 50% of its maximum value. The beauty of the method originally was not only its speed (the actual test takes less than 1–2 s for most solids), but the ability to use very small, 10–12 mm diameter and 1–2 mm thick samples. This advantage became a problem for some composite materials, when such small samples are considered non-representative of a larger body. The introduction of a system for large samples (30 mm diameter and up to 5 mm thickness), described later, has alleviated this limitation to a great extent [8]. Nevertheless, the need for specific heat capacity data remained the main limitation for this method in generating thermal conductivity data. While it was found that with careful experimental techniques, one could actually calibrate the response of the detector that generates the signal for the curve, the procedure itself imposed new limitations of its own. The process, simple in principle, involves testing a sample of known heat capacity first and then the unknown, and comparing the magnitude of the two resultant curves. Obviously, for such a differential computation the experimental variables (environment, electronics, energy pulse, etc.) must stay constant for both the sample and the reference. In reality this was, and still is a serious problem for any instrument that can test only one sample at a time. It is easy to see that even the best-case scenario (testing the reference over a temperature range, waiting for system to cool down, and then testing the unknown), will make close duplication of conditions nearly impossible. In real operations, the reference test may be several days, weeks, or months apart from the test performed on the unknown sample, allowing drifts and changes to severely impact the quality of data.

A major improvement was achieved with the introduction of multiple sample testing systems, where the unknown and the reference are tested side-by-side [9] in true differential fashion. The results typically improved from the ±7 to ±10% accurate data for single sample conventional systems, to ±2 to ±3% for the multiple sample configuration [10]. Since both specific heat capacity and thermal diffusivity are now measured in the same test, thermal conductivity is directly obtained in real time, with a priori knowledge of density.

Experimental

The work was done using a FlashLine™ 3000 Thermal Properties Analyzer. This devise accepts three samples side-by-side, inside its infrared heated furnace chamber. The atmosphere within the chamber is controlled to protect the samples from oxidation or other similar effects. Below the infrared furnace is the high speed Xenon pulse source. The light from the Xenon tube is concentrated and fed into the chamber via a wave-guide terminating right below the central sample position. A forward and reverse translation of the sample holder brings the two outside samples into the central position for testing. Above this position and outside the furnace chamber is the liquid nitrogen cooled InSb infrared detector. The image of the top surface of the sample situated in the central position is projected onto the detector with the help of intervening infrared optics. The temperature of the sample under test is detected by a type K thermocouple located adjacent to it and in intimate contact with the sample holder.

Results and discussion

The uncertainty associated with specific heat capacity and thermal diffusivity determinations using this equipment have been shown to be better than ±3% at 95% confidence level [11], when using high conductivity, small size samples. To extend the qualification to low conductivity materials and with large size samples, Pyroceram 9606, an industry-accepted reference material for thermal conductivity, was tested along with Pyrex glass, reinforced nylon and PVC. Table 1 presents thermal conductivity data at room temperature obtained from results of concurrent thermal diffusivity and specific heat capacity measurements performed with the flash technique, combined with results of density measurements on these materials. For comparison, data from steady state measurements on the same materials using a guarded heat flow meter instrument is also presented.

Table 1 Thermal conductivity on selected solids at room temperature

Method	PVC	Thermal conductivity/$Wm^{-1}K^{-1}$		
		Pyrex	Pyroceram	Nylon(reinforced)
Steady state	0.38	1.125	3.98	0.38
Flash	0.39	1.130	4.02	0.39

The study was extended for Pyroceram 9606 up to 1000°C. Figure 1 shows the excellent agreement between the results of thermal diffusivity measurements performed on this material up to 1000°C, and published data [12].

In all cases, the measured thermal diffusivity values are very close to the recommended data, and the repeatability is shown to be better than ±2%.

Table 2 was generated using concurrently measured thermal diffusivity and specific heat capacity for Pyroceram 9606, and using density values derived from results of thermal expansion tests performed up to 1000°C.

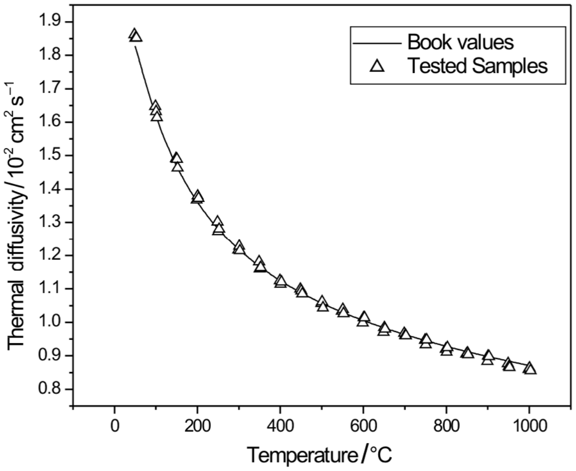

Fig. 1 Thermal diffusivity values of Corning Pyroceram 9606

Table 2 Thermal conductivity of Pyroceram 9606

Temperature/°C	Thermal conductivity/$Wm^{-1}K^{-1}$
100	3.66
200	3.46
300	3.35
400	3.09
500	3.05
600	3.06
700	2.95
800	2.85
900	2.81
1000	2.78

Conclusions

Thermal conductivity data at room temperature on selected materials is presented as a result of thermal diffusivity and specific heat capacity measurements using the flash technique, and compared with results of conventional steady state measurements on the same materials. High temperature thermal conductivity data obtained using the flash method on Pyroceram 9606 is also presented.

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The special chapter ends here.