APPLICATION OF THE LASER PULSE METHOD OF MEASURING THERMAL DIFFUSIVITY TO THIN ALUMINA AND SILICON SAMPLES IN A WIDE TEMPERATURE RANGE

by

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Paper presents results of measuring thermal diffusivity of translucent or partially transparent thin discs of non-metals such as alumina and silicon using most widely spread experimental technique, the standard laser pulse method. Difficulties in its application to such materials are discussed. The thermal diffusivity has been measured from room temperature up to 900 °C for alumina, and to 1200 °C for silicon. Obtained results are analyzed and compared with available literature data and existing recommended functions.

Key words: *thermal diffusivity, laser flash method, thermophysical properties, translucent materials, transparent materials, standard reference materials*

Introduction

The original laser pulse method of measuring thermal diffusivity proposed by Parker *et al*. [1] assumes ideal boundary and initial conditions, *i. e*. zero heat loss from the sample, infinitely short laser pulse, and uniform heating of the sample face. Thermal diffusivity is then obtained from analyzing the rear side temperature *vs*. time history, generally time of the half-rise of the temperature response. Simplicity of the method is marred in practice by difficulties of realizing these idealized conditions. Thanks to theoretical works of many researchers, the original concept has been gradually improved to account for real experimental conditions, making it the most popular technique responsible for over 80% of all thermal diffusivity results published within last few decades. A review of these contributions is given in [2].

The laser flash method can be used for many kinds of solid materials. The method implies the use of disk-shaped specimens, non-transparent both for the laser beam radiation and the radiation that comes from generated heat inside the specimen. In cases when specimen material is translucent or partially transparent for the spectrum of the laser beam radiation, in order to cut out the laser light absorption within the depth of the specimen a thin layer of some opaque solid material is coated at least on the incident specimen side.

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Objective of reported research was to demonstrate the application of standard laser flash method for thermal diffusivity measurements to laser-light translucent, and/or IR transparent materials. The paper reports results of thermal diffusivity measurements for both cases of mentioned transparent materials. Applied alumina (Al_2O_3) specimens were translucent for the laser beam radiation, while silicon (Si) specimens were partially transparent for IR radiation. Their small thickness made the task more difficult.

Experimental

The apparatus used in the measurements is described in detail in [3]. As a pulse energy source, it uses a pulse laser with a ruby rod 15.9 mm in diameter and 165 mm in length, operating at 694.3 nm, with the maximum pulse output energy of 30 J. Temperature rise of the rear specimen side was measured by InSb photovoltaic detector for specimen temperatures from 20 to 250 °C and by PbS photoconductive detector for temperatures from above 250 °C. Both detectors integrated the temperature response over the whole surface of the specimen rear side. The schematic of the apparatus is presented in fig. 1.

The corresponding data reduction has been preformed according to parameter estimation procedure described by Milošević *et al*. [4].

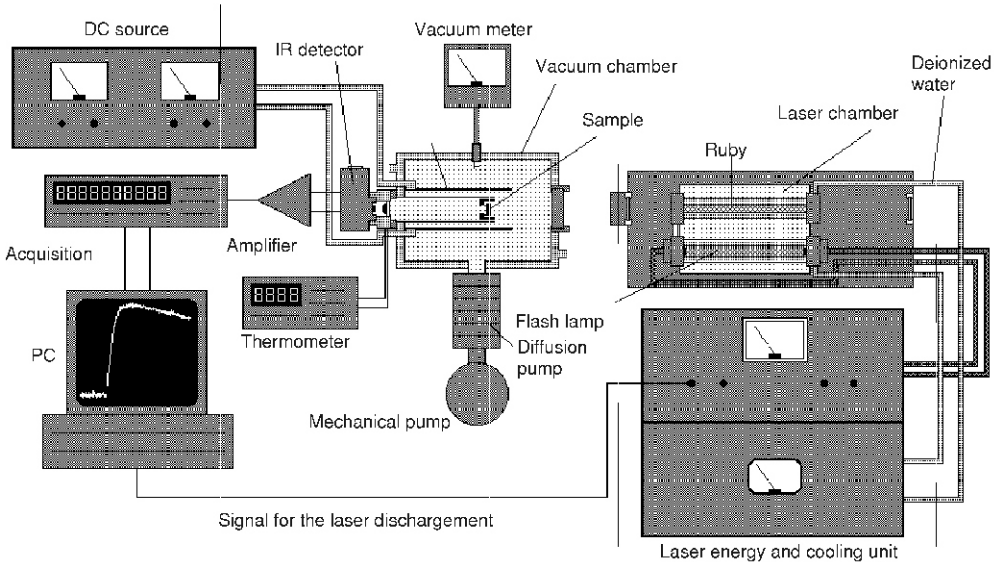


Figure 1. Schematic diagram of applied laser flash apparatus

Specimens and results

Both specimens were supplied by the UK National Physical Laboratory, Teddington, and were made from the highest grade of alumina and silicon rods. The specimen discs were machined to 10 mm in diameter and 1.01 mm for alumina, and 1.45 mm for silicon in thickness. The specimens were originally provided without any coating.

Alumina (Al_2O_3)

As uncoated alumina specimen was translucent to the ruby laser beam, it could not be used before further intervention. The coating was performed by a colloidal graphite spray and the thickness of a deposited layer was about 20 mm. In order to increase the emissivity of the rear specimen side (and thus the detectivity of transient temperature), the graphite deposition was also applied on the rear specimen side.

The coated alumina specimen was subjected to a series of measurements within a wide temperature range. In order to extend working life of the coating layer, which is affected by laser pulses, only few laser shots were fired and the same number of temperature responses was recorded at each reference temperature. After 900 °C, the graphite layer was damaged and experiments were ended.

Experimental results obtained on coated alumina specimen, given in tab. 1, represent average values at each reference temperature with corresponding uncertainty limits (coverage factor 2). Reported expanded uncertainties include data scattering and uncertainty proper to applied experimental technique.

Comparison of these results with other literature data [5-10] is given in fig. 2. As it can be seen, their agreement with the most of literature values is fair, especially at high temperatures.

Table 1. Experimental results of thermal diffusivity of alumina specimen coated by colloidal graphite

Temperature [°C]	Thermal diffusivity [10^{-6} m^{2}s^{-1}]	Uncertainty [%]
19	10.09	2.8
98	6.51	2.8
261	3.80	2.5
377	2.93	2.3
453	2.56	2.3
530	2.27	2.2
613	1.88	2.5
700	1.71	1.9
797	1.62	1.9
898	1.01	2.2

Silicon (Si)

In the first experimental run, temperature responses of uncoated silicon specimen were recorded from room temperature to about 1200 °C. Due to the silicon high transparency to infrared radiation generated by absorption of the laser pulse at the specimen front side, no useful responses were measured at temperatures below 550 °C. A gradual decrease of the material transparency to this internal radiation following further increase of the reference temperature is illustrated

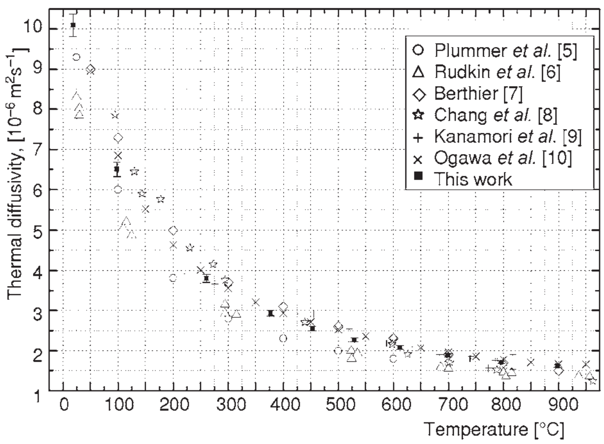
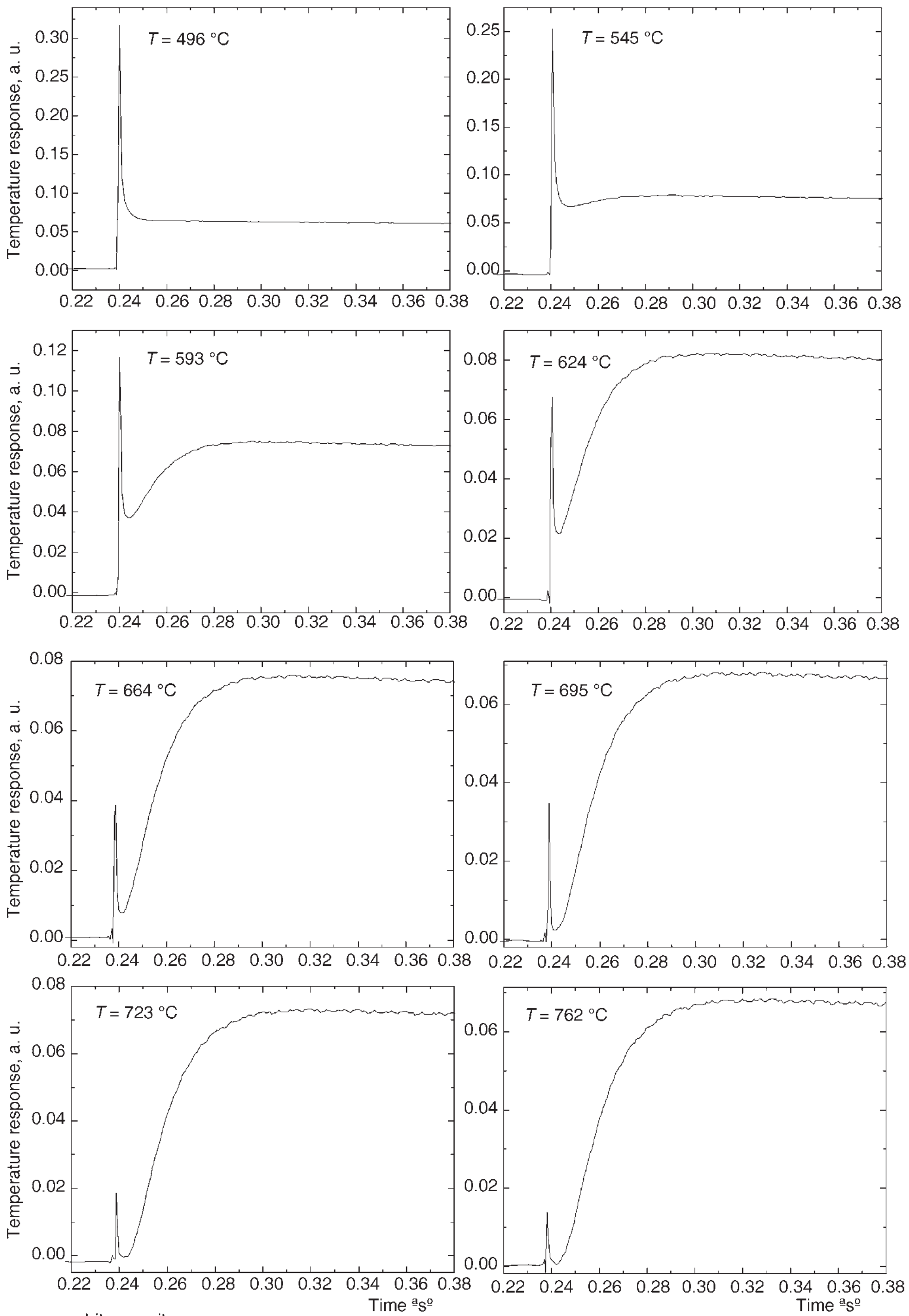


Figure 2. Thermal diffusivity of coated alumina specimen



a. u. – arbitrary units

Figure 3. Temperature responses of uncoated silicon specimen

in fig. 3. Above 650 °C, the radiative heat transfer through the specimen apparently becomes negligible in comparison to the conductive heat transfer.

During the first experimental run, at temperatures above 1150 °C thin-walled stainless steel tube which supports the sample holder started to evaporate, resulting in a deposition of a very thin metallic layer on the front specimen side. The second experimental run referred, therefore, to measurements on the same silicon specimen, but coated on its front side with thin metallic layer.

At high temperatures, responses of the specimen coated with the metallic layer were less deformed by the inside radiation heat transfer, resulting in better agreement between the values of thermal diffusivity obtained in the second run with the recommended values proposed by Touloukian *et al*. [11] (fig. 4). However, presence of coating had no effect at temperatures below 650 °C. The reason for this unexpected effect may be in wavelengths of the radiation of corresponding surface after the pulse absorption. While in the first run the radiation of absorbed pulse energy was emitted by the surface of pure silicon, in the second was released by another material, *i. e.*, by a stainless steel coating. Thus the transparency level of the silicon specimen may be dependent on different wavelength spectra of emitted internal radiation.

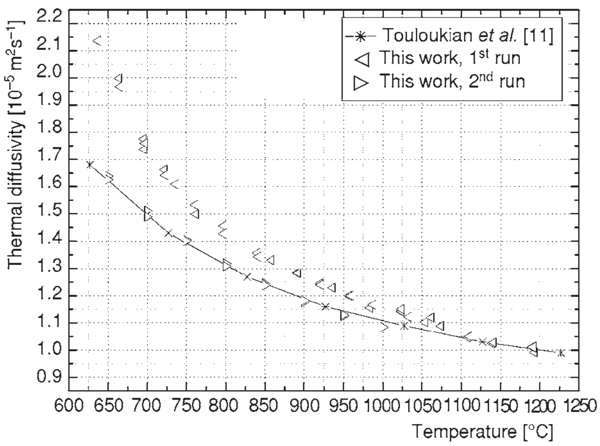


Figure 4. Thermal diffusivity of uncoated silicon specimen (1st run) and silicon specimen coated with a metallic layer (2nd run)

In the third, final experimental run, the measurements were performed with the silicon specimen coated by thin layer (20 mm) of colloidal graphite on both specimen sides. In contrast to first two runs, temperature responses recorded in this case were useful in the whole temperature range. The graphite layers persisted up to about 900 °C, but measurements could be continued at higher temperatures because the silicon specimen remained non-transparent to internal radiation.

Mean values of thermal diffusivity obtained in the third run, and corresponding expanded uncertainties (coverage factor 2) are given in tab. 2. These results are also presented in fig. 5, together with recommended values and other literature data [12-15]. It can be seen that at high temperatures our values agree very well with the recommended ones, but at temperatures below 600 °C they lie about 10% lower. The reason may be in applied graphite layer, which increases the characteristic time of temperature response and thus reduce computed thermal diffusivity value. Having in mind that the characteristic half-rise time at room temperature was 4 ms, in contrast to 30 ms at 1200 °C, it is evident that the influence of the graphite layer should be more pronounced at lower temperatures. For correction of this effect, another heat diffusion model with known thermophysical properties of graphite layer must be applied.

Table 2. Experimental results of thermal diffusivity of silicon specimen coated by colloidal graphite

Temperature [°C]	Thermal diffusivity [10^{-6} m^{2}s^{-1}]	Uncertainty [%]	Temperature [°C]	Thermal diffusivity [10^{-6} m^{2}s^{-1}]	Uncertainty [%]
19	80.8	3.2	646	15.4	2.0
104	52.7	2.6	707	14.1	2.1
266	32.1	2.5	797	12.9	2.4
361	25.4	2.1	902	11.4	2.5
453	21.5	2.2	982	10.8	2.1
536	18.3	2.2	1109	10.5	2.2
599	17.1	2.3	1195	9.77	2.8

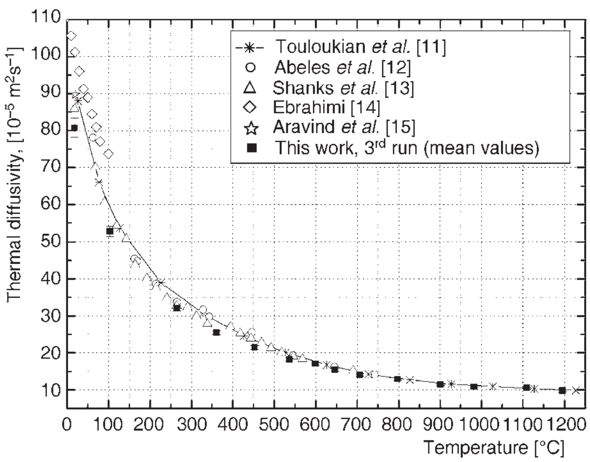


Figure 5. Thermal diffusivity of silicon specimen coated with colloidal graphite (3rd run)

Conclusions

In comparison to other literature data, this study made evidence that the laser flash method, regularly used to the thermal diffusivity measurements of opaque solid materials, might be applied to properly coated translucent or partially transparent solid materials as well.

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