MEASUREMENT OF ADVANCED MATERIALS USING THE FLASH METHOD

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Abstract

The paper deals with the laser flash method which is the most popular experimental method for measurement the thermal diffusivity. Although the flash method was primary developed for measurement of homogeneous isotropic materials, the method was successfully applied for an estimation on advanced and more complex materials. The paper summarizes present-day knowledge related to an application of the flash method to measure semitransparent materials, anisotropic media, layered structures, composites and thin films.

Key words: advanced materials, thermal diffusivity, flash method

1 Introduction

The flash method was first described by Parker et al [1] of the US Navy Radiological Defense Laboratory. In the flash method the front face of a small wall-shaped sample receives a pulse of radiant energy coming either from a laser, or a flash lamp. The thermal diffusivity value is computed from the resulting temperature response on the opposite (rear) face of the sample. The simple ideal analytical model of the flash method is based on the thermal behavior of a homogeneous opaque thermally insulated infinite slab uniformly subjected to a short heat pulse of radiant energy over its surface. The model assumes:

a) the sample is homogeneous and isotropic, and the thermophysical properties and the density are uniform, constant and invariant with temperature within the experimental conditions,
b) the sample is thermally insulated – there are no heat losses from the slab surfaces,
c) the heat pulse is uniformly distributed over the slab surface, and it is absorbed by a layer of material which is very thin in comparison to the thickness of the sample,
d) the heat pulse is instantaneous, its duration is negligible compared to the thermal response of the slab.

The one-dimensional heat flow occurs across the slab under these assumptions. The shape of the rear face temperature rise curve contains the information about the thermal diffusivity of the material. The conventional way to calculate the thermal diffusivity is based on the knowledge of the half time $t_{0.5}$ – the time in which the rear face temperature rise reaches one half of its maximum value. The thermal diffusivity a is calculated from the expression

$$a = 0.1388\frac{e^2}{t_{0.5}} \quad , \tag{1}$$

where e is the sample thickness.
Several other original data reduction methods (the algorithm for computing the thermal diffusivity from experimental data) in the flash method have appeared in the literature so far. They differ either in the analytical mathematical models used, or in the way the measured experimental rear face temperature vs. time data by theoretical curve, are respectively compared. The survey of the existing data reduction methods can be found elsewhere [2,3].

2 Semitransparent Materials

Particular difficulties occur if the measured material is transparent for the electromagnetic waves at the working wavelength of the laser or if the sample is transparent at the wavelength of the infrared temperature detector that is used. Then the detector measures temperature rise that is influenced by direct radiation across the sample. There are principal discussions among experts about how to interpret this effect. It can be viewed as the disturbing phenomenon that influences the measurement of the thermal diffusivity - the property that describes the heat conduction in the body. The other approach is to take the radiation into account and consider the thermal diffusivity as a parameter that consists of the sum of the radiation part as well as of the heat conduction part.
Coating the sample surfaces with a thin layer of opaque material usually decreases the direct radiation across the sample. The data reduction then requires using the two- or three-layered model.
More about how to deal with problems in measurement of semitransparent materials [4]-[10], or combined radiative/conductive heat transfer in heterogeneous semitransparent materials [11], can be found in the literature.

3 Anisotropic Media

To measure simultaneously the axial thermal diffusivity (across the sample) as well as radial thermal diffusivity (parallel to the front and rear surfaces) of an anisotropic material of cylindrical symmetry the flash method with radial heat flow was proposed [12] [13]. This technique is known as the radial flash method. The method consists of irradiating the central sample front face circular area of the radius r_p smaller then the sample radius r_s. If the temperature response is monitored at the rear face at two different locations both axial and radial thermal diffusivities are simultaneously deduced from the recorded experimental temperature vs. time data [12] – [16].
A generalization of the flash technique for measurements of orthotropic materials with three mutually orthogonal thermal diffusivities for finite and semi-infinite solids has been elaborated, too [17].

4 Layered Structures

It has been demonstrated by several authors that the flash method primarily developed for the measurement of the thermal diffusivity of homogeneous materials is suitable for

studying layered structures. It is essential that the materials boundaries are flat and parallel to the sample front and rear surfaces. If there are no heat losses from the radial surface one-dimensional heat transfer occurs across the sample and then by analyzing the temperature rise vs. time data any thermophysical property value (thermal diffusivity of one layer, or the thermal contact resistance) can be computed.
The basic theory for the two- and the three-layered composites was originated in [18]. Here the simple adiabatic models are described. The proposed data reduction method is based on the half-rise point similar as in the case of the original approach to homogeneous samples. In the case where the composite is formed from capacitive layers - layers whose material has a very high thermal diffusivity so that there is no thermal gradient across the layer, the theory described in [19] should be used. These models are suitable when analyzing composites that consist of a combination of metal and a poor conductor. Practical aspects of estimation on layered composites are given in [20] [21].
More general models, described in [22] - [24], cover measurements in the wider range of outer boundary conditions, and materials (higher temperatures, poor conductive layers) and more sophisticated problems, that are connected with complex layered materials. The theory can be applied to studying the functionally gradient materials - materials with a certain profile of thermophysical properties as given in [25] [26].
It is important to note that the determination of the thermal diffusivity of
a component (or the contact thermal resistance between two layers) in layered systems is a dependent measurement. An estimation of the thermal diffusivity of one layer or the thermal contact resistance requires besides the knowledge of other relevant properties (the density, the heat capacity and the thickness of components) to know the thermal diffusivity of the remaining layer(s). Errors in measurement of these additional properties are propagated through the data reduction and result in inaccuracy of the thermal diffusivity calculation. These effects have been investigated in two-layered materials and special conditions for reliable determination of the thermal diffusivity have been given [23] [27]. The paper [27] also discusses the concept of the apparent thermal diffusivity obtained from the experimental data and the mean thermal diffusivity which has physical meaning related to the thermal resistance.
Recently a new analytical theory for two-dimensional heat conduction through the two-layered material was developed, that enables an estimation of the thermal diffusivity and the thermal contact resistance using the data obtained by two-dimensional flash method [28].

5 Composites

The theory of the flash method assumes that the measured material is homogeneous. Therefore the thermal diffusivity measurements of various composites (fine weave, dispersed, fiber reinforced, etc.) are performed under the assumption that the composite material behaves as a homogeneous medium. This assumption may be acceptable if the scale of the microstructure is generally far smaller than the size of the sample and this is obviously true for sufficiently thick samples [29]-[32]. In the case of longitudinal heat flow through composites with fiber reinforcements (especially directionally reinforced composites) and if the fiber length is comparable with the sample thickness, there may be serious problems if such materials are considered to be homogeneous in the flash diffusivity measurement. These problems have been analyzed by various authors [33] -

[37], and it has been shown, that such assumptions may lead to inadequate results, like time- and sample-thickness-dependent diffusivities. If a fiber reinforced composite has to be measured, the homogeneous medium assumption can be applied, as long as the sample thickness is much larger than the fiber diameter, the fiber volume fraction is as large as possible, and the fiber and matrix are in perfect thermal contact, i.e. interfacial thermal conductance is large.

6 Thin Films

The laser flash method has been employed to measure very thin materials (thin films). When such a material is investigated in the transverse direction some effects, which may be ignored for thick samples, should become important because of the limitation of the measuring system. Heating and sensing speed originate experimental restrictions. The phenomena - the finite heat pulse duration effect, the inertia and the non-linearity of the temperature detector, the response time of the measuring system, and the finite absorption depth effect [38] [39] limit the sample thickness.

Special methods for measurement of the in-plane thermal diffusivity for thin materials having large length to thickness ratio were proposed. They are based on heating of the sample on one end by a pulse [40], periodic heat flow [41] [42] and the step heating [43]. The temperature is monitored at the opposite end of the sample serves for the thermal diffusivity estimation.

Acknowledgement

Authors wish to thank the Slovak Science Grant Agency for the financial support under the contract 1/9415/02.

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