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Material Characterisation

Thermophysical characterization of polymers according to the temperature using a periodic method

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A B S T R A C T

In this paper, we developed a method for measuring thermophysical properties of materials as a function of the temperature using a periodic thermal excitation. This method was tested on three polymers (PVDF, HDPE, PA6). The comparison of specific heat capacity values obtained by this method with those of the literature and those obtained by differential scanning calorimetry is used to validate this measurement method for this kind of material. Moreover, the obtained variation of thermal conductivity and thermal diffusivity of Polyamide 6 versus temperature was compared to measurements performed using a commercial Hot-Disk™ device.

1. Introduction

Contrary to other types of materials such as ceramics [1] and metals [2,3], we have little information about the evolution of thermophysical properties of polymers versus temperature. Knowledge of thermophysical properties of polymeric materials, such as thermal conductivity, thermal diffusivity and specific heat are of fundamental importance for scientists and engineers, in both processing stages and use of materials [4].

Polymers are, from a thermal point of view, insulating materials. In bulk form, their thermal conductivity lies between 0.1 and 0.5 $W.m^{-1}.K^{-1}$ at room temperature [4]. This conductivity depends mainly on the polymer composition, the organization of macromolecular chains (polymer amorphous or semi-crystalline) and state (glassy or rubbery). Heat transfer can be improved by adding conductive materials (metals or ceramics for instance). On the contrary, processing of polymer foams highly reduces heat transfer and allows obtaining a thermal conductivity close to the one of the air. Finally, as for most of the physical properties of polymers, the temperature has an influence on the values of the thermal conductivity.

A large number of experimental techniques have been developed for determining thermophysical properties of a material [2,3,5–21]. They can be divided into three main categories depending on the excitation mode: static, periodic or transient (see Table 1). For each of these methods, the measurement protocol allows obtaining one or two thermophysical parameters among thermal conductivity (k), thermal diffusivity (a), thermal effusivity (e) and specific heat (C_p). At this stage, it is important to recall that the knowledge of two of these four parameters allows computing the two other ones, if the sample density (ρ) is known, using the set of equations below:

$$\begin{cases} a = \frac{k}{\rho . C_p} \\ e = \frac{k}{\sqrt{a}} = \sqrt{k.\ \rho .\ C_p} \end{cases} \quad (1)$$

Methods based on a measurement in a static regime allow obtaining only the thermal conductivity of the investigated sample. Moreover, measurement durations with these techniques are quite long (sometimes several days) especially if different temperatures are considered. Some other methods in transient or periodic regimes allow also obtaining only one thermophysical parameter (thermal effusivity, thermal diffusivity, thermal conductivity or specific heat). In that case, the determination of the other thermophysical parameters requires an additional measurement and/or the knowledge of a second thermophysical parameter.

Some existing methods are based on an optical or an acoustic excitation of the sample, thus requiring specific devices such as laser or flash sources and optical detectors such as thermopiles or infrared sensors in the case of photothermal methods. Moreover, in that case, some additional properties of the material, like its emissivity for instance have to be known. A bad knowledge of such parameters can lead in some cases to systematic errors on the estimation of the thermophysical parameters.

At this time, among existing techniques, the non-steady-state-techniques are increasingly used [22], mainly because they allow a

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Table 1
Overview of measurement methods of thermophysical properties (k: thermal conductivity; a: thermal diffusivity; e: thermal effusivity; C_p: heat capacity).

Measurement techniques	Measured thermophysical property	Steady state/transient/periodic method	Excitation type	Reference
Guarded hot-plate	k	Static	Conduction, constant power	ISO8302 (1991); Degiovanni [5]
Bar method	k	Static	Conduction, constant power	Degiovanni [5]
Angström method	a	Periodic	Conduction, sinusoidal flux	Angström [6]
Fin method	a	Transient	Conduction	Degiovanni [7]
Hot-wire method	k	Transient	Conduction, constant power	ISO8894; Degiovanni [5]
Hot-wire with separated source and sensor	k, e	Transient	Conduction, constant power	Krapez [8,9]
Asymmetric guarded hot-wire	k, e	Transient	Conduction, constant power	Krapez [8,9]
Isotherm probe	e	Transient	Contact between two solids	Balageas et al. [10]; Krapez [8,9]
Hot plane	e	Transient	Conduction, constant power	Krapez [8,9]
Hot strip	e	Transient	Conduction, constant power	Krapez [8,9]
Dynamic plane source	a, e	Transient	Conduction, constant power	Karawacki [2]; Krapez [8,9]
Extended dynamic plane source	a, e	Transient	Conduction, constant power	Krapez [8,9]
Hot plane with separated heater and sensors	a, e	Transient	Conduction, pulse energy density fixed	Pierrus [11]; Krapez [8,9]
Hot disk	k, a	Transient	Conduction, variable power	Gustaffson [12]
3ω method	k	Periodic	Conduction, variable power	Cahill [13], Bigg [35]
(Front face) flash method	a, e	Transient	Flash	Parker [14]; Degiovanni [5]
(Rear face) flash method	a	Transient	Flash	Krapez [8,9]
Photothermal radiometry	e	Transient	Radiative exchanges, constant power	Krapez [8,9]
Photoacoustic method	a, e	Periodic	Modulated laser	Veleva [3]; Krapez [8,9]
Photopyroelectric method	a, e	Periodic	Modulated laser	Dardalat [15,16]; Thoen [17]; Preethy Menon [18]; Delenclos [19]; Baldears-Lopez [20]; Pittois [21]
DICO	k, a	Periodic	Conduction, modulated temperature	Boudenne [26,27]
DSC	C_p	Temperature ramp	Conduction, variable power	Wunderlich [36]