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Non-contact methods for thermal properties measurement

B. Abad[a], D.-A. Borca-Tasciuc[a,b], M.S. Martin-Gonzalez[a,*]

[a] IMM-Instituto de Microelectrónica de Madrid (CNM-CSIC), Isaac Newton 8, PTM, E-28760 Tres Cantos, Madrid, Spain
[b] Department of Mechanical, Aerospace and Nuclear Engineering, Rensselaer Polytechnic Institute, 110 8th Street, Troy, NY 12180 USA

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ABSTRACT

Many of the renewable and sustainable energy technologies employ novel nanomaterials. For instance, thermal storage and thermoelectric conversion are in constant progress due to the emergence of new structures such as carbon-based materials, bulk nanostructures, 2D novel materials or nanowires. Thermal properties play a significant role to all these energy technologies as key parameters to evaluate the performance and efficiency of those materials in the final device. Understanding the effects of nanostructuring on thermal properties becomes critical, since a reduction in the thermal conductivity due to increased phonon scattering at interfaces is usually expected. Therefore, the determination of the thermal properties remains a critical aspect of material development effort, and measurement techniques are continuously developed or improved. Among those, non-contact heating methods are of importance since they bypass a frequent source of errors characteristic to contact-based thermal measurements, namely the thermal contact resistances, which can be dominant in nanoscale materials. Non-contact heating techniques are usually based on photothermal phenomenon, where heating is generated typically by incident radiation. This paper reviews non-contact heating measurement methods, providing an overview of basic principles for measurement along with associated theoretical model necessary for data reduction and their main applications. The techniques are categorized as time domain and frequency domain techniques, where the thermal response of the sample under study is analyzed as a function of time and frequency, respectively. Both types of methods study the transient response of the sample from a pulsed or modulated heating, and typical measurement output is thermal diffusivity. In addition, other non-contact techniques are also discussed, such as those based on steady-state response, from which the thermal conductivity is directly obtained, or those using AFM probe in the non-contact mode. Finally, main advantages and disadvantages of these techniques are summarized along with their associated uncertainties.

1. Introduction

Among technologies included in the renewable and sustainable energy portfolio, thermoelectric energy conversion, thermal energy storage and hydrogen storage are of high interest for several reasons as discussed next. Sustainable energy approaches involve optimization of energy efficiency to achieve thermal comfort [1,2] or thermal regenerators which act as heat exchangers [3]. Furthermore, in mainstream energy production techniques about two-thirds of the energy is lost as waste heat [4]. Thermoelectric energy conversion has been tapping into this source fueled by the development of more efficient thermoelectric materials such as complex structures, nanocomposite materials with tunable thermal properties or low-dimensional materials like semiconductor nanowires [5]. Similarly, storing thermal energy has been garnering attention recently due to the development of new nanomaterials with enhanced thermal properties, including 2D materials such as graphene [6], phase change materials (PCS), carbon-based materials or metal foams composites and nanomaterials [7]. In addition, great efforts are focused on hydrogen storage materials for stationary energy storage applications where new structures like Mg-materials make it possible to improve this technology [8,9].

A key performance parameter of nanomaterials developed for aforementioned energy technologies is thermal conductivity. For example, low thermal conductivity is required in thermoelectric applications, to limit the heat leakage between high and low temperature reservoirs. Thus, low-dimensional structures such as bulk nanostructured materials, thin films or superlattices along with nanowires have been broadly studied [5,10]. On the other hand, high thermal conductivity is necessary in hydrogen storage materials in order to promote the heat transfer required for the hydrogen sorption kinetics, but also in thermal energy storage applications in order to avoid the suppression of the energy charging and discharging rates [11]. Therefore, highly conductive materials such as carbon nanotubes or graphene are frequently introduced to enhance the thermal properties

* Corresponding author.

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of these materials. Thus, being able to engineer thermal conductivity is an important part of the material development effort, and this process is informed by experimental thermal conductivity measurements. Measurement of thermal properties of nanostructured materials is typically challenging and different techniques are required depending on the dimensionality and properties of each structure. The challenges can be especially difficult when attempting to measure materials with one dimension in the nanoscale range, such as thin film or nanowires. For instance, during the measurement of thermal conductivity or diffusivity, the specimen is locally heated and the thermal response is recorded at various locations away from the heated spot. The thermal property is then extracted by interpreting the experimental temperature data based on the theoretical analysis of the heat transfer inside the material. Depending on the particularities of the experimental setup, the heat transfer can be affected by heat losses (which become dominant at large surface to volume ratio) or thermal contact resistances (such as between the thermal probes/electrodes and the material), which may become dominant in nanoscale samples and difficult to predict accurately.

In general, the techniques employed for thermal properties characterization can be categorized based on the time dependence of the thermal response as steady-state or transient methods as seen in Fig. 1(a).

For instance, in steady-state characterization, the heat is applied uniformly across a sample of uniform cross-section. The temperature is measured at both ends of the sample and the thermal conductivity is determined by applying a one-dimensional heat transfer model, knowing the sample dimensions [12–17]. On the other hand, transient methods study the thermal response as a function of time when heating the material [18,19]. The experimental setup and the data reduction of the steady-state techniques are usually simpler than the transient techniques. However, the steady-state measurements are more time-consuming and are extremely sensitive to heat losses through convection, radiation, and conduction.

Thermal properties characterization techniques are also classified depending on the heating procedure as contact and non-contact methods. In the former, the sample is heated by direct contact where the heating source can be either a microprobe as in Scanning Thermal Microscopy [20–23], a thin-film electrical resistor deposited directly onto the surface of the sample as in 3ω [24,25], a bolometer [26], as in pulse heating [27] and micro-bridge methods [21,28], or an external resistor in the case of hot disk [29] or equivalent steady-state setups. These techniques have been used for measuring a variety of structures such as bulk, thick and thin films or nanowire arrays, although when

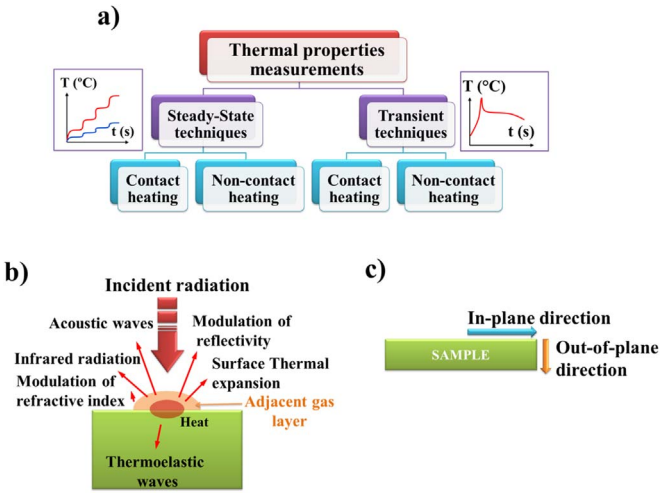

Fig. 1. (a) Classification of the thermal conductivity and diffusivity measurement techniques, (b) thermal effects associated with incident radiation absorbed by a surface and (c) main measurement directions in a sample.

measuring thermal conductivity of single nanowires, microchips are typically employed [30–34]. A main common disadvantage of these techniques is the thermal contact resistance between the heat source and the sample. This makes it challenging to perform an accurate measurement especially when measuring nanostructures like nanowires or thin films, whose low thermal resistance can be easily surpassed by contact thermal resistance. On the contrary, dimensions of the bulk samples can be tuned in order to have a larger thermal resistance and render the effect of contact resistance negligible. Besides the thermal contact resistance, the complex microfabrication of the thin film electrical resistor patterning or the microchip is the major drawback in the sample preparation for many of the contact measurement techniques.

In contrast, non-contact methods are typically based on photothermal phenomenon where heating is produced by an absorbed incident radiation. Thus, contact resistance from a heat source is avoided. These techniques are also of interest due to the simplicity of sample preparation. However, since it is difficult to establish accurately the amount of heat absorbed in the sample, the non-contact methods generally make use of a transient or a modulated response, and the transport property determined experimentally is thermal diffusivity or effusivity. The thermal diffusivity, α, indicates how fast a material responds to a temperature change and it is related to the thermal conductivity, κ, by,

$$\kappa = \alpha \cdot C_p \cdot \rho \tag{1}$$

where ρ is the density and C_p is the specific heat. On the other hand, the thermal effusivity, e, describes the capability of a material to exchange heat with its surroundings and is given by,

$$e = \sqrt{\kappa \cdot C_p \cdot \rho} \tag{2}$$

Therefore, the main disadvantage of these methods arises from the necessity of knowing both density and specific heat in order to backup thermal conductivity. However, these properties can be determined by other methods such as differential scanning calorimetry (DSC) for specific heat measurement or pycnometry for density measurements. Oftentimes, when characterizing thermal transport properties of nanostructures, the density and specific heat are considered similar to bulk [35,36]. Yet, this assumption should be used with caution as it could lead to errors, since changes in phonon dispersion can affect the specific heat [37].

Although most of the non-contact techniques are based on optical heating, the measurement approach may be widely different depending on the thermally-sensitive phenomena employed in the detection scheme. Thermal emission [38], thermal expansion of the sample [39], refractive index changes [40], and acoustic waves [41] are some examples of the effects that can be caused by heating the sample optically and used for temperature measurement as schematically represented in Fig. 1(b). Most of the techniques make use of 1D heat transfer models, which allows for obtaining the thermal conductivity in the out-of-plane direction, i.e., perpendicular to the surface of the sample. However, in-plane thermal conductivity, which corresponds to the direction parallel to the surface of the sample, as depicted in Fig. 1(c) can also be measured. This fact is of great importance allowing for an understanding of thermal anisotropy in materials.

Regardless of the detection approach used, these techniques can be divided into two main groups: time-domain and frequency-domain [35]. The time-domain methods are based on the analysis of the thermal response as a function of time, such as for example, the decay in sample temperature after pulse heating. In contrast, the frequency-domain methods make use of an incident modulated radiation, which generates a periodic signal whose amplitude and phase dependence on frequency is analyzed.

Besides these categories, additional methods employing non-contact optical heating for thermal conductivity measurement involve the