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Heat-loss modified Angstrom method for simultaneous measurements of thermal diffusivity and conductivity of graphite sheets: The origins of heat loss in Angstrom method

Yuan Zhu

State Key Laboratory of Optoelectronic Materials and Technologies, School of Physics and Engineering, Sun Yet-Sen University, Guangzhou 510275, China

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ABSTRACT

Angstrom method is a steady-state measurement for thermal diffusivity α using ac heating. Since thermal conductivity λ is a better-known quantity, measured diffusivity α is sometimes transformed into λ based on relation $\lambda = C_v\alpha$ using recorded or DSC measured C_v. However, Angstrom method itself is principally possible to extend to specific heat measurements, yet the accuracy is not promising due to the complexity of heat loss. Here we present a modified method for simultaneous measurements of thermal diffusivity and thermal conductivity with high accuracy by taking heat loss into account. A linear heat loss term m^2T is introduced into the diffusion equation and the thermal conductivity λ can be directly measured instead of specific heat. The measured thermal properties of commercial graphite sheets agree well with their nominal value. The origins of m^2 are also discussed. m^2 can be divided into amplitude independent and dependent part. From the basics of radiation and convection, the first-order radiation and convection comprise the amplitude independent part, while the dependent part includes higher order (dominated by second-order) radiation. Although the amplitude independent part agrees well with the extrapolated value of m^2 at zero amplitude, the second-order radiation cannot fully cover the measured amplitude dependent part. This discrepancy is further explained by floating temperature baseline variation due to residual heat during heat oscillation.

1. Introduction

The use of steady-state temperature oscillations for thermal diffusivity α measurements was first stated by Angstrom in 1861 and is generally known as Angstrom method [1,2]. It has not been properly replaced by other techniques (e.g. Laser flash apparatus [3]) for many years due to its mathematical clearance and convenience of installation. Numerous variations of Angstrom method have been developed to obtain material thermal properties in different shape and condition (e.g. Wagoner [4], Kosky [5–7], Cerceo [8], Topolnicki [9], Roetzel [10], Baughn [11] and Smalc [12]). Among these variations, simultaneous measurement of the diffusivity α and volumetric specific heat C_v is an important branch. Since thermal conductivity λ is a better-known quantity, measured diffusivity α values are sometimes transform into λ based on relation $\lambda = C_v\alpha$. Before simultaneous measurements were well developed, this can only be done whether using recorded volumetric specific heat C_v [4,12,13], or using complementary instruments such as Differential Scanning Calorimeter (DSC) [12].

In principle, it is possible for Angstrom method to derive C_v by alternating heater power or oscillation frequency. In practice, however, it was considered of low accuracy due to the complexity of the heat loss analysis [14]. Basically, Angstrom method is independent to heat loss for diffusivity α measuring, while it is indeed heat-loss dependent for specific heat C_v measuring. Sullivan et al. [15] accepted the inaccuracy in the absolute C_v value, while argued that the changes in C_v can be more precisely measured by measuring frequency-dependent temperature amplitude. Yet no heat-loss induced error was discussed in their work. There are other attempts on adding heat loss term into the thermal diffusion equation. Since the effect of heat loss varies for different sample dimensions and measuring models, understanding the origins of the heat loss in different instrumentals and modeling them properly are crucial to the validity and accuracy of data reduction and analysis.

For slab or plate samples clapped by heater or detector at both sides, chopped laser or thermoelectric module is the most used heating sources. Choices depend on whether in-plane or cross-plane properties are wanted. For in-plane diffusivity or conductivity, the 2D infinite plane model is used by heating the

E-mail address: azalea.zhu@gmail.com

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sample center with a small laser spot [13]. For cross-plane diffusivity or conductivity, the 1D thermal wave model is used by heating the entire area of one surface of the sample using TE (Thermoelectric) module [16]. For 2D infinite plane model, Visser et al. [13] introduced dimensionless variable δ into the solution to account for convective and radiative heat loss from the surface to sample. The authors estimated that $\delta < 0.1$, which they claimed might be neglected for their case.

For strip or bundle samples, the heating power is usually mounted on one end of the sample and the 1D semi-infinite model is commonly used due to the much longer sample length compared to thermal wavelength [4,17,18]. Its heat loss mostly lies in the sample surfaces that are much larger than the sample-heater or sample-holder interfaces. This is quite different a scenario from the slab or plate sample, whose heat loss mostly lies in the interfaces. For the measurement of carbon fibber bundles, Wagoner and his co-workers [4] introduce the heat loss term *BDT* into 1D thermal diffusion equation diffusivity, where *B* is heat loss parameter, *D* is diffusivity and *T* is temperature. They assumed the heat loss was a *T*-linear term and the parameter was proportional to diffusivity. Their assumption was not verified in their work since they focused on diffusivity and did not mention heat loss any further. To the best of our knowledge, no work has simultaneously extracted diffusivity and specific heat from a suitable series of measurements for strip samples and needless to say the heat loss effect on the measurements.

This work will focus on the strip samples and investigate the heat-loss effects on thermal diffusivity and specific heat/thermal conductivity measurements. Graphite sheets are used as typical strip samples since it is an anisotropic material with a in-plane diffusivity much larger than cross-plane. Angstrom method is a wide-accepted way to measure its in-plane diffusivity. Once the specific heat or conductivity can also be extracted from the same set-up, its practical utility aggrandizes. In Section 2, the heat-loss included Angstrom model is developed by introducing a heat loss term to the thermal diffusion equation. Derivation of this modified equation make it possible to simultaneously extract thermal diffusivity and thermal conductivity from a series of experiments. Based on this mathematic modification, experiments and new data analysis approach are provided in Section 3. The amplitude and measuring distance dependence of the measured diffusivity and conductivity are revealed in Section 4. The origins of heat loss are analyzed in very detail in Section 5. Uncertainty and error estimation are attached in Supplementary Information.

2. Theory

2.1. General

With heat loss, the one dimensional heat diffusion equation can be rewritten as

$$\frac{1}{\alpha}\frac{\partial T}{\partial t} + m^2 T = \frac{\partial^2 T}{\partial^2 x} \tag{1}$$

where α is thermal diffusivity [m^2/s], T is temperature [K], and m is the coefficient of surface heat loss. Although heat loss includes conduction ($\sim T$), convection ($\sim T$) and radiation ($\sim T^4$), we only use the homogeneous T-linear term m^2T as an approximation for math convenience. The validity of this approximation will be demonstrated later.

When a sinusoidal heat wave is sent down to the sample in shape of stripe from one end ($x = 0$) (as shown in Fig. 1), the solution of Eq. (1) is also in waveform of

$$T(x,t) = A + B(x)e^{i\omega t} \tag{2}$$

where i is the unit imaginary, ω is angular frequency of the heat wave, and A is a constant indicative of the baseline for the temperature oscillation. One can also set $A = 0$ for mathematical concise so that the T should be taken as temperature deviation from the baseline rather than the absolute temperature. The temperature variation induced by the waveform heating has a complex amplitude $B(x)$.

Substituting $T(x,t) = B(x)e^{i\omega t}$ into Eq. (1), the second order ordinary differential equation for $B(x)$ gives

$$B_{xx}(x) - \left(\frac{i\omega}{\alpha} + m^2\right)B(x) = 0 \tag{3}$$

where $B_{xx}(x)$ is the second derivative of $B(x)$ with respect to x. The general solution of Eq. (3) is

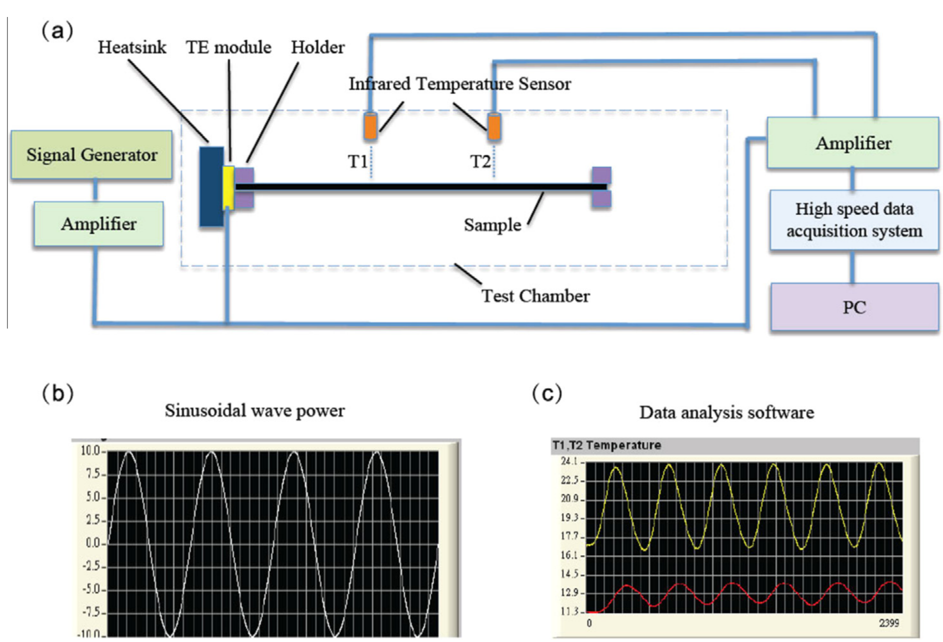

Fig. 1. Schematic set-up of the Angstrom method (a) and the snapshots of the heat power wave (b) and measured temperature wave (c).