Measurement of thermal diffusivity by a modification of the Angstroem's method using thermally short specimens

by Alberto Muscio*

**DIMeC – Dipartimento di Ingegneria Meccanica e Civile*
Università di Modena e Reggio Emilia
Via Vignolese 905/B, 41100 Modena, Italy
Phone +39-059-2056194, Fax +39-059-2056126, E-mail: alberto.muscio@unimore.it

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Abstract

A modification of the Angstroem's method for measurement of thermal diffusivity is presented. The Angstroem's method relies upon the steady-periodic propagation of temperature waves along the specimen. The diffusivity is recovered from combination of phase shift and amplitude decay of the waves under the hypothesis of virtually semi-infinite specimen, that is neglecting the reflection of temperature waves at the end of the specimen.

In this work, a condition is exploited in which the specimen is not considered semi-infinite. Testing of a large set of materials is made possible, as well as recovering the diffusivity value from either amplitude decay or phase shift, independently. This is often impossible when a condition of 'thermally semi-infinite' specimen must be achieved. A procedure to estimate the diffusivity is developed from the mathematical model of the 'thermally short' specimen and it is subsequently validated by numerical simulation. Finally, the results of preliminary experiments are presented.

1. Introduction

Thermal diffusivity is a property difficult to measure, especially for materials such as pure metals, crystalline oxides, semiconductors or layered composites. Those have high value of the diffusivity and are often available as plane thin specimens. Moreover, they are frequently anisotropic, with diffusivity through the thickness different from that in parallel to the plane of the specimen. The standard ‘flash method’ allows measuring the diffusivity through the thickness, but not in the specimen plane. It also requires a rigorous preparation of the experiment. Other techniques have been studied, but so far an alternative standard has not been established.

A good candidate for a new standard test method is the technique proposed by Angstroem in 1861. It relies upon the steady-periodic propagation of temperature waves. The diffusivity is recovered from phase shift and amplitude decay of the waves along the specimen. More specifically, Angstroem employed a long bar of small cross-section. One end of the bar was subjected to a periodic change of temperature, being alternately heated by a current of steam and cooled by a current of cold water for equal time intervals. After a few cycles the temperature field settles down to a steady-periodic state, which was monitored by Angstroem at selected points along the bar.

The Angstroem’s method was rediscovered and modified in several manners since the beginning of the past century, as modern laboratory instrumentation more and more allowed an effective generation and monitoring of the temperature waves. A more accurate assessment of phase shift and amplitude decay of the temperature waves was recently made possible by computerized data-acquisition and data-processing systems. A significant advancement was also given by the introduction of non-contact temperature sensors and thermographic instruments.

Contact heaters, electron bombardment, light beams being cyclically modulated or periodically shielded by a moving mask, and chopped laser beams were largely employed. Sources with a positive net heat input, however, produce a large stationary gradient of temperature along the specimen. This may influence heat transfer between specimen and surrounding medium, causing significant discrepancy from the theoretical models. A great number of cycles is also needed to achieve steady-periodic conditions. Above all, it is difficult to produce a sinusoidal temperature oscillation and the analysis of data is made difficult. Therefore, contact sources based on the Peltier effect were also employed. These allow one to alternate heating and cooling stages, thus obtaining a null net heat input and temperature oscillations about the ambient value.

The Angstroem's method and its modifications are based on the assumption of virtually semi-infinite specimen, that is on neglecting the reflection of temperature waves at the end of the specimen opposite to the thermal source. This makes simple the mathematical model through which the diffusivity is estimated, but it requires relatively long specimens and long time cycles to achieve a satisfactory signal-to-noise ratio. Nonetheless, exploiting a condition in which the specimen is not considered semi-infinite permits to test a larger set of materials and, above all, to recover the diffusivity value from either amplitude decay or phase shift of the temperature waves, independently. This is often impossible when a condition of 'thermally semi-infinite' specimen is achieved, as the effects of heat transfer between specimen and test ambient must be filtered by combining amplitude decay and phase shift data.

In this work the mathematical model for the 'thermally short' specimen condition is presented. More specifically, the model is obtained by solution of the Fourier's equation of heat transfer. A procedure to estimate the thermal diffusivity is then developed from the model and subsequently validated by numerical simulation. Finally, the results of preliminary experiments are presented, obtained by means of an apparatus for measurement of the in-plane diffusivity of thin specimens. The apparatus, integrated under the Labview programming environment for virtual instrumentation, is based on a Peltier thermoelectric source with closed loop control and an infrared camera for temperature measurement along the specimen.

2. Mathematical model and estimate procedure

The mathematical model for the 'thermally short' specimen condition was developed by solution of the Fourier's equation of heat transfer. A harmonic temperature oscillation was assumed at the end of the specimen in contact with the thermal source as it is allowed by a Peltier device. Heat transfer between the specimen and the test ambient by convection and radiation was also considered.

The non-dimensional temperature field is described by the real part of Eq. (1).

$$\vartheta(\xi,\tau) = Re\left\{\frac{\left(\gamma + i\frac{1}{\gamma}\right)cosh\left[\left(\gamma + i\frac{1}{\gamma}\right)(\Xi - \xi)\right] + Bi_L\, sinh\left[\left(\gamma + i\frac{1}{\gamma}\right)(\Xi - \xi)\right]}{\left(\gamma + i\frac{1}{\gamma}\right)cosh\left[\left(\gamma + i\frac{1}{\gamma}\right)\Xi\right] + Bi_L\, sinh\left[\left(\gamma + i\frac{1}{\gamma}\right)\Xi\right]} exp(i2\pi\tau)\right\} \tag{1}$$

where ξ=*x*/L and Ξ=*X*/*L* are the space position *x* and the specimen length *X* made dimensionless by the thermal diffusion length *L*, γ takes into account heat transfer between side surfaces of the specimen and test ambient, Bi_L takes into account heat transfer at the specimen end opposite to the thermal source, and τ=*t*/t_0 is time *t* made dimensionless by the time-cycle of the temperature oscillation t_0. *L* includes the thermal diffusivity α to be estimated and it is defined as follows:

$$L = \sqrt{\frac{\alpha t_0}{\pi}} \tag{2}$$

Neglecting heat loss (but not wave reflection) at the specimen end opposite to the thermal source, i.e. assuming Bi_L=0, the temperature field is described by a relationship in the form

$$\vartheta(\xi,\tau) = \Delta\vartheta(\xi)cos\left[2\pi\tau + \varphi(\xi)\right] \tag{3}$$

where non-dimensional amplitude decay and phase shift are given in Eqs. (4)-(5):

$$\Delta\vartheta(\xi) = \frac{\sqrt{\left\{cosh\left[\gamma(2\Xi - \xi)\right]cos\left(\frac{1}{\gamma}\xi\right) + cosh(\gamma\xi)cos\left[\frac{1}{\gamma}(2\Xi - \xi)\right]\right\}^2 + \left\{sinh\left[\gamma(2\Xi - \xi)\right]sin\left(\frac{1}{\gamma}\xi\right) + sinh(\gamma\xi)sin\left[\frac{1}{\gamma}(2\Xi - \xi)\right]\right\}^2}}{cosh(\gamma 2\Xi) + cos\left(\frac{1}{\gamma}2\Xi\right)} \tag{4}$$

$$\varphi(\xi) = -arctan\left\{\frac{sinh\left[\gamma(2\Xi - \xi)\right]sin\left(\frac{1}{\gamma}\xi\right) + sinh(\gamma\xi)sin\left[\frac{1}{\gamma}(2\Xi - \xi)\right]}{cosh\left[\gamma(2\Xi - \xi)\right]cos\left(\frac{1}{\gamma}\xi\right) + cosh(\gamma\xi)cos\left[\frac{1}{\gamma}(2\Xi - \xi)\right]}\right\} \tag{5}$$

The diffusivity is obtained by fitting the time evolution pattern of temperature along the specimen. More specifically, amplitude decay and phase shift are evaluated for each position along the specimen and then fitted separately with Eq. (4) and Eq. (5) to recover the diffusivity value. In the experimental practice, Equation (5) is expected to produce better results as phase measurements are less affected by non-homogeneous emissivity of the specimen surface. A non-linear least square fitting procedure has been implemented in the Matlab programming environment, considering sets of temperature data as large as permitted by current thermographic cameras, acquired along several time cycles.

The estimate procedure has been validated by fitting temperature data produced by numerical simulation. Excellent results have been obtained, achieving an accurate estimate of the diffusivity even when artificial noise with gaussian distribution has been added to the numerically generated data, with average intensity comparable to current uncooled thermographic cameras. Preliminary experiments on an AISI-304 specimen, using phase shift data, have also given interesting results.