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MEASUREMENTS OF THERMAL DIFFUSIVITY AT SEMITRANSPARENTS MEDIA

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Abstract. *To determine thermal diffusivity of semitransparent media at temperature the backside temperature rise of a cylindrical sample is subjected to short pulsed heat flux. This sample is thus heated to certain temperature and temporal temperature variation can be used to measure the thermal diffusivity. The Maximum A Posteriori (MAP) criterion is used for the parameter estimation procedure.*

Keywords: thermal diffusivity, flash method, semitransparent media.

1. INTRODUCTION

The flash method introduced by Parker *et al.*(1961) has been used for thermal diffusivity measurement of opaque. As is known, the front side of the sample is irradiated by short pulse. The temperature at the backside is monitored using thermal detection. It is possible to use the transient response of materials to identify one or more thermal characteristics of the medium. In the case of interaction of thermal radiation with an absorbing, emitting and scattering medium, the simultaneous conduction and radiation heat transfer problem must be considered. Compared with the direct measurement of thermal conductivity, the advantages of this method are simple sample geometry, easy sample preparation and small sample size, as well as applicability for a wide range of diffusivity values an excellent accuracy and reproducibility. Also, because very litle time is needed for a single measurement, a wide range fo temperature can be covered in a short period of time.

The transient heat transfer for an absorbing and emitting medium was investigated by Ping *et al.*(1991) and Andre and Degiovanni (1995). The scattering property was considered by Hahn *et al.*(1997) The aforementioned articles concern the flash method to determinate thermal or apparent diffusivity. In order to investigate the case of transient coupled conductive - radiative heat transfer in semitransparent medium is necessary the knowledge of intrinsic properties such as thermal capacity (ρ.c), refractive index (n), scattering coefficient (σ), absorption coefficient (κ).

The aim of this study consists in the thermal diffusivity measurement of semitransparent media from the knowledge of transient temperature taken at the backside of the sample. The maximum A posteriori estimation (MAP) is used for the minimisation of general non-linear function.

2. DIRECT PROBLEM

We consider transient simultaneous conduction and radiation in gray, absorbing, emitting, and isotropic scattering, plan - parallel. At time $t_0 = 0$, the surface at z = 0 is subject to a heat pulse on the entire face, while the surface at z = e is kept at a prescribed temperature.

Other characteristics of the model are : a global heat exchange coefficient *h* is include to describe the convective-radiative heat losses; the temporal boundary condition is expressed by converting the heat pulse into a temperature jump for the first half-elementary volume of the medium. The surfaces are gray and the physical of medium properties are constant. A schema of the physical system and coordinates is shown in "Fig. 1". The energy equation is taken as

$$\frac{\partial^2 T}{\partial z^2} - \frac{1}{k}\nabla.\mathbf{q}^{\mathbf{r}} = \frac{\rho c_p}{k}\frac{\partial T}{\partial t}, \qquad 0 < z < e, \qquad t > 0 \tag{1}$$

with boundary conditions

$$k\frac{\partial T}{\partial z} = h(T - T_e) + q^r_{net1} \qquad z = e,\ t > 0 \tag{2}$$

$$-k\frac{\partial T}{\partial z} = h(T - T_e) + q^r_{net2} \qquad z = 0,\ t > 0 \tag{3}$$

Initial Conditions

$$T_i = T_e + \frac{q_{flash}}{\rho c_p \varepsilon}; \qquad 0 < z < \varepsilon, \qquad t=0 \tag{4}$$

$$T_i = T_e; \qquad \varepsilon < z < e, \qquad t=0 \tag{5}$$

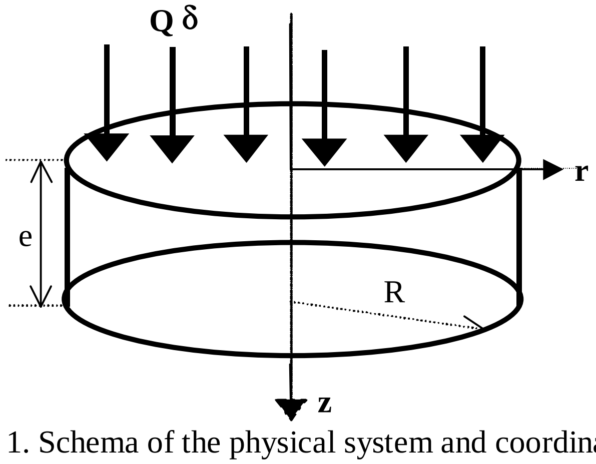

Fig. 1. Schema of the physical system and coordinates.

Where T is the temperature, T_e is the equilibrium temperature, k the thermal conductivity, ρc_p the thermal capacity, q^r_{net} the net heat flux from the surface and $\nabla.\mathbf{q}^{\mathbf{r}}$ the divergence of the radiation heat flux vector. They are, respectively, determined from

$$q^r_{net} = \int_{4\pi} \mathbf{n} \cdot \mathbf{\Omega}\, I(\mathbf{\Omega}) d\Omega \qquad (6)$$

and

$$\nabla . \mathbf{q^r} = \kappa\left(4n^2 \bar{\sigma} T^4 - \int_{4\pi} I(\Omega) d\Omega\right) \qquad (7)$$

where n is the refractive index, κ the average absorption coefficient, $\bar{\sigma}$ Stefan-Boltzmann constant, and n the unit surface normal vector.

The net radiative heat flux q^r_{net} and the divergence of the radiation heat flux vector $\nabla . \mathbf{q^r}$ must be determined from the solution of the radiative transfer equation. In this case, the equation of transfer for radiation intensity along a path s is given by Özisik (1973) as

$$\frac{dI(s)}{ds} + \beta I(s) = \beta S(s) \qquad (8)$$

where I(s) is the intensity at location s, the extinction coefficient and source term are

$$\beta = \kappa + \sigma \qquad (10)$$

$$S(s) = \kappa I_b(s) + \frac{\sigma}{4\pi} \int_{4\pi} I(s') p(\mathbf{\Omega}', \mathbf{\Omega}) d\mathbf{\Omega}' \qquad (11)$$

In "Equation (11)", $\mathbf{\Omega}$ is the unit vector pointing in the radiation direction. The integration over the solid angle $\mathbf{d\Omega}$ is done over the intensity at s considering all possible directions.

Integrating "Eq.(8)" along a path s we get the integral form of the radiation transfer equation as follows

$$I(s) = I(s_0, \mathbf{\Omega_0}) \exp[-\beta(s - s_0)] + \int_{s_0}^{s} S(s') \exp[-\beta(s - s')] \beta ds' \qquad (12)$$

The first term on the right in "Eq. (12)" is the contribution of the radiation intensity coming from the boundary at location s_0. The radiation intensity at location s is equal the one at the boundary reduced by a factor $e^{[-\beta(s-s0)]}$ due to the absorption and to the scattering along the integration path. The second term represents an increase due to the term source distributed along of the integration path.

The "Equations (1 - 5) and (12)" provide the mathematical formulation for the radiation-conduction interaction direct problem in a one-dimensional medium. An iterative process is needed to solve the problem, because the energy equation, "Eq. (1)", involves the radiation intensity, while the equation of radiation transfer, "Eq. (12)", requires the temperature profile.

3. METHOD OF SOLUTION

The S_n or the discrete ordinates method (Fiveland, 1987) is used to solve the integral form of the radiative transfer equation or the radiation part of the problem. The variation law for the radiation intensity incident on the medium at s_0 and at the location s along path to each discrete direction is established. The semitransparent medium is homogeneous and isotropic, gray, by a emitting, absorbing and linear-anisotropic scattering bounded emitting, diffusely reflecting wall, as show the "Figure 2".

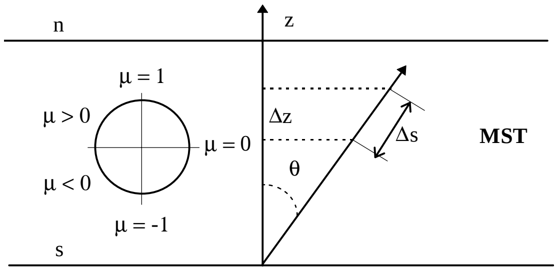

Figure 2 - physical model by radiation problem.

The discrete ordinates form law of the variation law of the radiation intensity is obtained by evaluating "Eq.(12)" at each of the discrete direction and replacing the integral term into "Eq.(11)" by numerical quadrature to give

$$\int_{\Omega'=4\pi} I(s,\Omega')p(\Omega',\Omega)d\Omega' = \sum_{m'=1}^{N} I_{m'm}p_{m',m}w_{m'} \tag{13}$$

where: $w_{m'}$ are the weights associated with each direction $\Omega_{m'}$, $p_{m'm}$ the values of the phase function corresponding to incident direction $\Omega_{m'}$ and to scattering direction Ω_m and $I_{m',m}$ the radiation intensity in the discrete direction $\Omega_{m'}$.

In this work, the technique proposed by Carslson and Lathrop (1966) is applied to calculated the quadrature points and weights.

To determine the distribution of the radiation intensity. The variation law of the intensity must be established (Da Silva, 1997). This law is established while following the propagation of the radiation to each direction Ω_m, in every element from a point where the intensity is known. So, one determines the value of the intensity after a distance Δs. These law is given in the chosen coordinate system what permits us to determine the distribution of the intensity in the physical domain without using the equation of classic finite differences . So, if we assume that I_0 is uniform on boundary and S does not vary with z in the distance Δs, then we can integrate "Eq.(12)" to find

$$I_m = I_{0,m} \exp[-\beta\Delta s(z)] + S_m\{1 - \exp[-\beta\Delta s(z)]\} \tag{14}$$

As we can see from this equation, the radiation intensity vary along the path s. Even in this case, we can define the average at the distance Δs by

$$\bar{I}_m = \frac{1}{\Delta s}\int_0^{\Delta s} I_m(s)ds \tag{15}$$

In the specific case of the one-dimensional geometry, the distance Δs can be expressed in cartesian coordinates by

$$\Delta s = \frac{z - z_0}{\mu_m} \tag{16}$$

now, let us introduced the "Eq. (16)" into "Eq. (14)". Then we get

$$\bar{I}_m = I_{0,m} \exp\left[-\beta\left(\frac{z - z_0}{\mu_m}\right)\right] + \\ + S_m\left(1 - \exp\left[-\beta\left(\frac{z - z_0}{\mu_m}\right)\right]\right) \quad (17)$$

Using the expression given by "Eq. (17)" into "Eq.(15)", we find

$$\bar{I}_m = \left(I_{0,m} - S_m\right)\left(1.0 - \exp\left[-\beta\left(\frac{z - z_0}{\mu_m}\right)\right]\right) \cdot \\ \cdot\left(\frac{\mu_m}{\beta(z - z_0)}\right) + S_m \quad (18)$$

This expression provides the average radiation intensity along the distance Δs. This average intensity permits us to calculate by iterative process the source term, and therefore the divergence of radiative flux that will be introduced in the resolution of the energy equation.

When the distribution of the radiation intensity is known, an solution to the energy equation for the temperature field in medium resulting from the conduction-radiation iteration and boundaries conditions is determined. The control volume approach is applied to obtain the finite difference form of the energy equation. Then, the non-linear finite difference problem is solved iteratively by the Newton-Raphson. Once the temperature field is determined, it is compared with the guess value used for radiation part of the problem, and the process is repeated until a specified convergence criterion is achieved. The "Fig. 3" shown theoretical backside thermograms for a specific example.

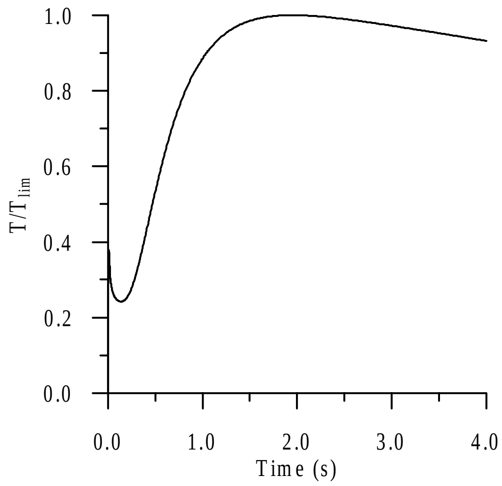

Figure 3 - Exact temperature of the backside temperature rise obtained for theoretical model.

4. INVERSE PROBLEM

Inverse heat conduction-radiation problem is similar to the above direct problem except that the thermal conductivity, thermal capacity and extinction coefficient are considered as unknown. Experimental backside temperature history of a semitransparent sample is available. Then, utilising measured temperature data and the exact temperature data can start the inverse problem. The inverse conduction-radiation problem is then solved by minimisation the error between the experimentally obtained surface temperature history and the predicted temperature at backside of the sample. The minimisation is accomplished by the Maximum A Posteriori estimation (MAP).

5. PARAMETER ESTIMATION METHOD

5.1 Choice of the criterion, model of measurement errors

Apart from a limited number of techniques such as Neural Network (Narenda et al., 1990) or Kalman filter (Scarpa et al. 1993), most of the parameter estimation procedures involve a criterion, composed of one or two objective functions, that is being extremized. Usually the functional, **S(β),** is quadratic but a Bayesian approach (Idier et al., 1996) can lead to other forms.

$$S(\beta)=\left[\mathbf{Y}-\eta(\beta)\right]^{\mathbf{T}}\mathbf{W}\left[\mathbf{Y}-\eta(\beta)\right] \quad + \quad \left[\mu-\beta\right]^{\mathbf{T}}\mathbf{U}\left[\mu-\beta\right] \tag{19}$$

where **Y** is the vector of measurements (n x 1), **W** and **U** are matrices that depend on the type of estimator. The dimensions of **W** and **U** are (n x n) and (p x p), respectively.
For Ordinary Least Squares (OLS) **W=I** and **U=0.**

The Maximum Likelihood (ML) criterion is obtained with $\mathbf{W}=\Psi^{-1}$ and **U=0**, where **Ψ** is the covariance matrix of the measurement errors. Usually, it is difficult to determine the covariance of all measurement errors but possible to determine their variances. If the variances are not constant then the ML method gives more weight to the measurements that have the smallest variances.
The Maximum A Posteriori estimation (MAP) uses not only information on the measurement errors but also on the unknown parameters: $\mathbf{W}=\Psi^{-1}$, μ are the a priori values of β and $\mathbf{U}=\mathbf{V}^{-1}$**.** **V** is the covariance matrix of the prior parameters. μ and **U** can either come from previous set of experiments or can be more subjective. The MAP criterion is such that the first component guaranties, to some extent, the fidelity of the solution to the measurements and that the second component satisfies some properties known a priori. One can see that if **V** is small and **Ψ** is large then, unless n is great, the information given by the measurements will barely change the prior values. Consequently the MAP method must be used with caution if the prior information is too subjective (the results will correspond to the belief of the investigator).

5.2 Choice of the extremisation method

There are many methods for the minimisation or maximisation of general non-linear functions, among these are exhaustive search, simplex exploration, Gradient methods (Gauss-Newton, conjugate gradient, Levenberg-Marquart), Iterative minimisation (Alifanov et al. 1995), adjoin method (Jarny et al. 1991), ... which are more or less sophisticated. The choice, which can lead to discussion that never ends, depends on the number of parameters to be estimated and to some extent on the structure of the criterion. Most of these methods are available in computational library (ISML, Numerical Recipes).

The Gauss-Newton method is one of the simplest and most appropriate methods when the number of unknown parameters is not large say 20. It specifies direction and size of the correction to the parameter vector. The principle is to find the vector **β=b** such that all the first derivatives of the model with respect the parameter is simultaneously equal to zero.

For linear-in-parameter problems, **b,** the estimated vector of the unknown β is given by

$$\mathbf{b} = \mu + \left(\mathbf{X}^{\mathbf{T}}\mathbf{W}\mathbf{X} + \mathbf{U}\right)^{-1}\mathbf{X}^{\mathbf{T}}\mathbf{W}\left(\mathbf{Y} - \mathbf{X}\mu\right). \tag{20}$$

which for the OLS method, reduces to

$$\mathbf{b} = \left(\mathbf{X}^{\mathbf{T}}\mathbf{X}\right)^{-1}\mathbf{X}^{\mathbf{T}}\mathbf{Y} \tag{21}$$

"Equation (20)" shows that the a priori values, μ, are based on the differences between the measurements, **Y**, and the model, **η=Xμ,** calculated with μ as input. This equation also shows how **Ψ** and **V** influences the estimated parameters.

For non-linear-in-parameter problems, **X=X(β),** and an iterative procedure must be used. With the superscript (*k*) as a counter of the iteration, an equation similar to "Eq. (20)," is obtained

$$\beta^{(k+1)} = \beta^{(k)} + \left[\mathbf{X}^{T(k)}\mathbf{W}\mathbf{X}^{(k)} + \mathbf{U}\right]^{-1}\left[\mathbf{X}^{T(k)}\mathbf{W}\left(\mathbf{Y} - \eta^{(k)}\right) + \mathbf{U}\left(\mu - \beta^{(k)}\right)\right] \tag{22}$$

The iterative procedure starts with an initial guess, $\beta^{(0)}$, at each step the vector β is modified until:

$$\frac{\left|\beta_i^{(k+1)} - \beta_i^{(k)}\right|}{\left|\beta_i^{(k)}\right| + \xi} < \delta \text{ for } i = 1, 2, ..., p \tag{23}$$

δ is a small number that must be chosen by the investigator (typically 10^{-3}) and ξ (< 10^{10}) prevents overflow if $\beta_i^{(k)} = 0$.

For MAP method, $\beta^{(0)} = \mu$. For OLS or ML methods **U = 0**, thus the initial guess has no influence on the final result, it only changes the number of iteration. A good guess facilitates the convergence. It is highly recommended to start the estimation procedures with several sets of initial estimates to check that they all converge to the same value. When the matrix ($\mathbf{X}^{\mathbf{T}}\mathbf{W}\mathbf{X}$ **+ U**) is not well conditioned, modifications to the Gauss-Newton method such as Levenberg-Marquart method are recommended.

6. EXPERIMENTAL DEVICE AND RESULTS

The experimental thermogram was obtained in the LEMTA(Laboratoire d'Energétique et de MécaniqueThéorique et Appliquée). Following, the components of the experimental basic device used to flash experience is described (e.g. Figure 2).

- the uniform heat flux is delivered by four linear flash lamp with total surface of the $50mm^2$. the output permits him to deliver a quantity of energy around 20 kJ/m^2;
- the sample has the cylindrical shape to plane and parallel faces and the thickness can vary 1 to 20 mm and can be diameter of 10 to 50 mm;

- the system of temperature detection, it is about two needles - dopey P and N - of $FeSi_2$ in contact separated on the sample that forms an intrinsic thermocouple, the signal is amplified recorded then by an oscilloscope to numeric memory;
- The amplifier convert the current delivered in tension and amplifies the signal;
- the oscilloscope to numeric memory permits to record thermogram in 12 bits on 4000 points;
- the heating of the sample involve an airflow what is sustained by fan and electrical heaters. The hot air circulates in the annular space of the quartz wall whose internal surface heats the sample by radiation. A thermal insulation in fibbers of ceramics, envelop the furnace in quartz where the intern temperatures reach thus 1000 K.; and
- a pump permits to reach a level of pressure of 10-2 to 10-3 Mb. At the time of the utilisation vacuum, a porthole in quartz transmits the visible radiance of the flash.

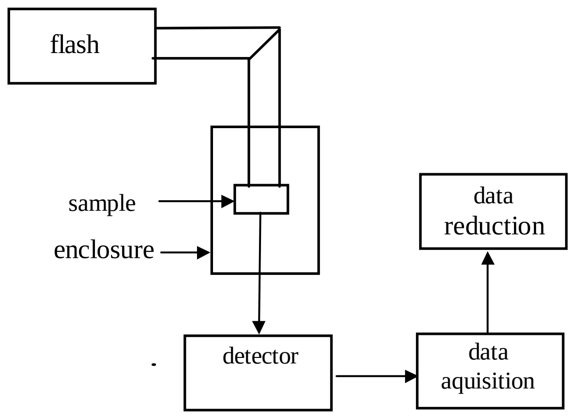

Figure 2 - System Schematic

The classical experience flash for the mesurements of the thermal diffusivity in case of an semitransparent media. The semitransparent media were ZnS or IRTRAN 2. The sample is then sandwiched between opaque layers and only the first internal reflection on the rear face is taken into account. The typical rear-face thermograms obtained by flash method and theoretical model recalculated with the diffusivity identificated is showed by "Fig. 3", showing the effect of heat losses and the residues.

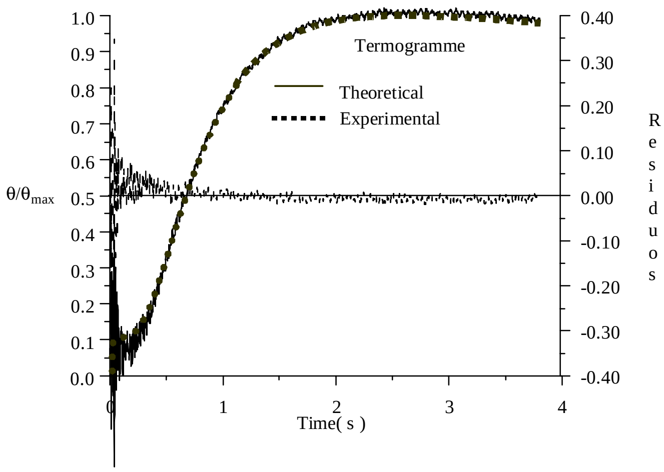

Figura 3 - The typical rear-face thermograms

The set of measurements is presented in table 1 for three different temperatures. In the experience were considered heat losses of sample, finite pulse duration, uniformity of the heat flux and non-variation thermal properties with temperature. The Maximum A Posterior estimation (MAP) criterion is used to estimate the parameters. The results show that the identificated diffusivity are good agreement.

Table 1 - Comparison of the Thermal diffusivity results obtained from two data reduction procedure.

temperature (K)	this work	LEMTA
298*	$8{,}26.10^{-6}$	$8{,}3.10^{-6}$
633	$2{,}98.10^{-6}$	$3{,}01.10^{-6}$
783	$2{,}32.10^{-6}$	$2{,}46.10^{-6}$
Step time: $\Delta t = 0.001$; 21 nodal points		
Interval of the identification : $0.001 \leq t \leq 0.6$		

7. Conclusion

The literature has showed that flash method for difusivity estimation is widely used not only for homogeneous or semi-infinite opaque sample, but also for a wide variety of materials. This work concern to a semitransparent media and the applicability of the method flash associated to the Maximum A Posterior estimation (MAP) criterion can be verified at very good precision.

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