Inverse Problems in Engineering: Theory and Practice
3rd Int. Conference on Inverse Problems in Engineering
June 13-18, 1999, Port Ludlow, WA, USA

HT12

STUDY OF GLUINGS BY PERIODIC METHOD : OPTIMAL DESIGN OF EXPERIMENTS

C.GERVAISE,
ENSICA 1 place Emile Blouin
31056 Toulouse cedex France

C.NOUALS
ENSICA 1 place Emile Blouin
31056 Toulouse cedex France

JJ.SERRA
DGA/DCE/CTA/LOT/GHF, BP6,
66125 Font Romeu cedex

C.CALDERAN
Université de Perpignan
52 avenue de Villeneuve 66860
Perpignan

S.BENET
Université de Perpignan
52 avenue de Villeneuve 66860
Perpignan

ABSTRACT

This paper presents the study of gluing thermal parameters by periodic methods. A classical gluing is featured by a thermal resistance in between two identical layers. A study of phaselag versus excitation gives an estimation of the gluing thermal parameters . A 1D model of simulation was the base for a ODE (Optimal Design of Experiments) global methodology. It is shown that front face configuration is well adapted for an NDC application. The optimal frequencies for the measurements of thermal resistance are calculated and consequences of errors on supposed to be known parameters are studied.

INTRODUCTION

Recently developed scientific and industrial applications use new materials such as multi layers coatings, thermal barriers and gluings in response to an increasing requirement of performance. It is then important to measure the thermal properties of these multi-layers materials in order to evaluate the performances of an application, to optimize the material build up or to predict its evolution versus time and wear.

Periodic methods used by Gervaise and al [1] [2] [3] can be applied to the study of multi-layers. A modulated laser beam heats the sample on its front face and the field of temperature is measured by radiometry on either the front heated face or the rear face. The temperature has a continuous component plus a periodic one whose period is identical to the heating beam one. Its fundamental harmonic is defined by its magnitude and its phaselag. A great interest of the periodic method is that the disturbed field area can be limited through the choice of the frequency. Dimension of this area is the diffusion length δ.

$$\delta = \sqrt{\frac{\alpha}{\pi f}} \qquad /1/$$

Phaselag records versus excitation frequency gives an estimation of multi layer thermal parameter : it is the identification by material transfer function.

A global methodology of identification of properties per material transfer function is proposed. An experimental bench dedicated to the study of multi-layer at millimetre scale using the periodic methods is described. A 1D modeling of experiments is presented and is adapted for a three-dimensional one is given. From 1D model, matrix X'X and sensitivity coefficients are used to Optimal Design of Experiments (ODE).

NOMENCLATURE

α : diffusivity m^2s^{-1}
CTR : Contact Thermal Resistance
δ : diffusion length m
ε : vector of measurement noise
f : frequency of excitation Hz
Φ : heat flux Wm^{-2}
φ : thermal phaselag rad or °
h : thermal loss coefficient $Wm^{-2}K^{-1}$
λ : conductivity $Wm^{-1}K^{-1}$
M : thermal modulus °K
NDC : non destructive control
R_c : thermal resistance $W^{-1}Km^2$

SBK : supposed to be known parameters
Si : sensitivity coefficient of phaselag versus parameter i
σi : standard deviation of parameter i
θ : vector of parameters
ω : pulsation of heat rads^{-1}
X : matrix of sensitivity coefficient
Y : vector of measurements

EXPERIMENTAL BENCH [2]

A cylindrical sample measuring some centimeters in diameter and some millimeters thick, is heated by a modulated laser beam on its front face. The size of the heating spot is typically 1 cm diameter disc. The field of temperature is measured on the front or rear face by infra-red radiometry. The laser beam has a frequency range from 0.01Hz to 2.5KHz and a power range from 0 to 50W. A constant concentrated solar flow monitor the average temperature of the sample (temperature of black body = 800°C). 800nm and 10.6µm optical wave length for heating laser beam and InSb and HgCdTe infrared detector are available and allow experiments on a large panel of material.

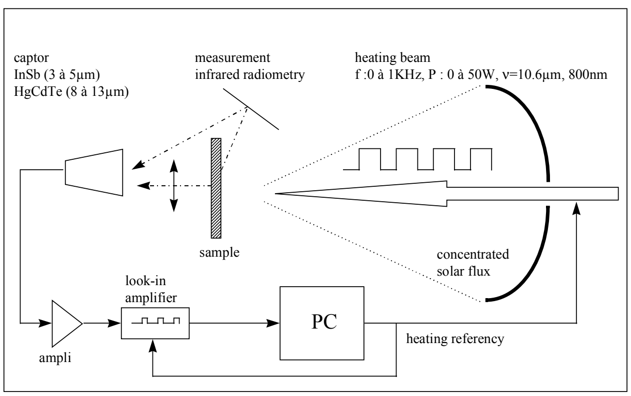

figure 1 : experimental bench

MODELLING

Simulations are dedicated to calculate the magnitude and phaselag of temperature field fundamental harmonic. One defines a complex temperature $T = Me^{j\varphi}$, M :magnitude, φ :phaselag.
A simulated sample is described :
- thickness e along axis X
- two homogeneous layers (α,λ)
- in between the layers, a CTR at depth p.

This sample is liable to radiative and convective losses from its both facs that can be modeled by a linearised coefficient h.
Equations associated with this geometry are the following ones:

$$\Delta T - \frac{j\omega}{\alpha} T = 0 \; for \; 0 \le x \le p \; and \; p \le x \le e \qquad /2/$$

$$-\lambda \frac{\partial T}{\partial x} = \Phi - h\,T \; for \; x = 0 \qquad /3/$$

$$-\lambda \frac{\partial T}{\partial x} = h\,T \; for \; x = e \qquad /4/$$

$$-R_c \lambda \frac{\partial T}{\partial x} = T(x = p_-) - T(x = p_+) \; for \; x = p$$
$$\lambda \frac{\partial T}{\partial x}(x = p_+) = \lambda \frac{\partial T}{\partial x}(x = p_-) \qquad /5/ \; /6/$$

As complex temperatures on the front face or the rear face are needed, a modeling by thermal quadrupole is developed. The thermal quadrupole associated with an homogeneous wall of thickness e, diffusivity α and conductivity λ heated by a wave pulse ω is:

$$Q_{wall} = \begin{vmatrix} ch(me) & -\frac{1}{\lambda m} sh(me) \\ \lambda m sh(me) & ch(me) \end{vmatrix} with \begin{bmatrix} T_s \\ \Phi_s \end{bmatrix} = Q_{wall} \begin{bmatrix} T_e \\ \Phi_e \end{bmatrix} \text{and}$$

$$m = \sqrt{\frac{j\omega}{\alpha}} \qquad /7/$$

The thermal quadrupole associated to a contact thermal resistance (CTR) R_c is :

$$Q_{CTR} = \begin{vmatrix} 1 & -R_c \\ 0 & 1 \end{vmatrix} with \begin{bmatrix} T_s \\ \Phi_s \end{bmatrix} = Q_{CTR} \begin{bmatrix} T_e \\ \Phi_e \end{bmatrix} \qquad /8/$$

Thermal transfer between the front face and the rear face is modeled by three quadrupoles put in series, two thermal quadrupoles associated to the homogeneous walls and an other one associated to the CTR. A matrix relation between Φe, Te, Φ_s, T_s is obtained. This relation added to the limits equations /3/ /4/ makes it possible to express T_e and T_s.
A 3D modeling [4] [5] with cylindrical symmetry around the heat laser beam axis can be derived from the 1D model. Thermal equations are written in three dimensional coordonees . The Hankel transform (versus $r = \sqrt{y^2 + z^2}$) is applied to each equation, the set of equations obtained is identical to the 1D modeling then one carries out an inverse numerical Hankel Transform to obtain final temperature field.

A « standard gluing» case of study used all along this paper is now defined : e=1mm, p=0.5*e, α=$10^{-5}m^2s^{-1}$, λ=$50Wm^{-1}K^{-1}$, R_c=$10^{-5}W^{-1}m^2K^{-1}$, h=$10Wm^{-2}K^{-1}$, e=1mm, p=0.5mm.

The two following figures give the distribution of phaselag for various values of R_c.

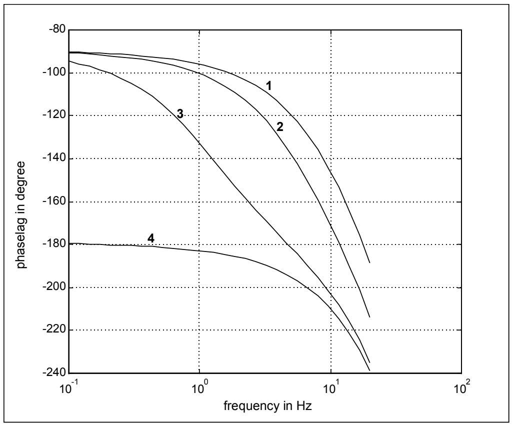

figure 2 : phaselag versus frequency at rear face « standard gluing case » with
1 : Rc=0 $W^{-1}m^2K^{-1}$, 2 : Rc=$10^{-5}W^{-1}m^2K^{-1}$,
3 : Rc=$10^{-4}W^{-1}m^2K^{-1}$, 4 : Rc=1 $W^{-1}m^2K^{-1}$

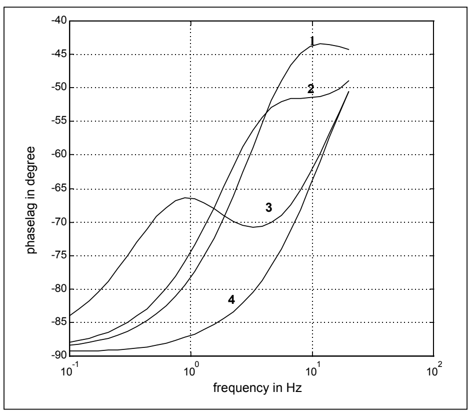

figure 3 : phaselag versus frequency at front face « standard gluing case » with
1 : Rc=0 $W^{-1}m^2K^{-1}$, 2 : Rc=$10^{-5}W^{-1}m^2K^{-1}$,
3 : Rc=$10^{-4}W^{-1}m^2K^{-1}$, 4 : Rc=1 $W^{-1}m^2K^{-1}$

For the rear face, the greater is the CTR the greater is the phaselag versus frequency. All the curves go through the same asymptote of equation $\varphi = -\frac{e}{\delta} = -\frac{e}{\sqrt{\alpha}}\sqrt{\pi f}$ at high frequencies.

For the front face, phaselag ranges from -45° for high frequency and 0° for low frequency if h is different from zero and -90° if h is null.

The median frequencies are dedicated to CTR influence.

OPTIMAL EXPERIMENT DESIGN [7] [6] [8] [9]

Both experimental bench and simulation models are used to study gluings.

The experimental design is defined by
- 2 parameters of sample's geometry : e and p
- 4 thermal parameters : λ, α, h and R_c ,
- an experimental setting : the frequency of excitation
- a choice between front or rear face experiment.

The goal of this chapter is to determine the optimal experimental settings and the best configuration to measure as accurately as possible the six associated parameters of a gluing.

The NDC point of view will be developed by choosing the best configuration to estimate the highest number of parameters in one experiment. A record of 30 points logarithmiquely distributed from 0.1Hz to 20 Hz is supposed to be available. This frequency area is the largest area reachable in the « standard gluing » case.

The metrology of CTR will be developed by choosing the best frequencies and by studying the influence of error on « Supposed to Be Known » parameters (SBK parameters). Optimal conditions minimize the variance of the estimated CTR.

For these two approaches, reduced sensitivity parameters, determinant and conditioning of X'X matrix, inverse of $1/\sigma^2$*X'X are used.

SENSITIVITY STUDY

Sensivity coefficient for rear face are given. S_i is the sentivity coefficient of phaselag versus the properties « i ».

$$S_i = p_i \frac{\partial \varphi}{\partial p_i}$$

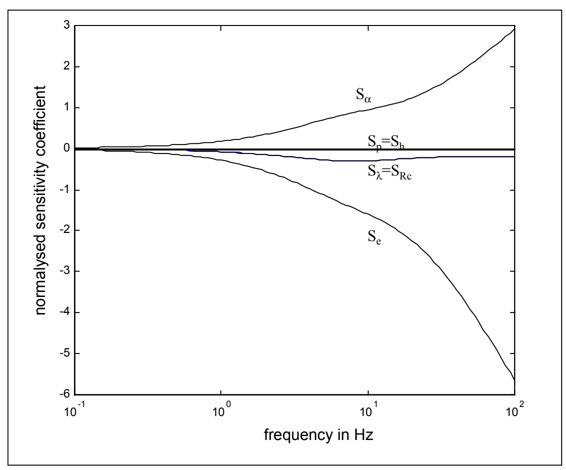

figure 4 : sensitivity coefficient at rear face « standard gluing case »

The sensitivity coefficients for all parameter are null towards the low frequencies since the phaselag tends towards a constant value.

Two parameters (α,p) present a sensitivity which tends towards infinite value for the large frequencies with an asymptotic value of the type $S_e = -2S_\alpha = -\frac{e}{\delta}$ in accordance with the spherical model.

One musts point out the local correlation between e and α.

Two parameters (Rc, λ) are correlated and present an medium level of sensibility with a global maximum.
Two parameters (h, p) have no sensitivity on the area of calculation. for p, the configuration p=e/2 causes this low level and for h the area of studied frequencies is too high for the phaselag to be sensitive to the heat loss.

Sensivity coefficient at front face are given.

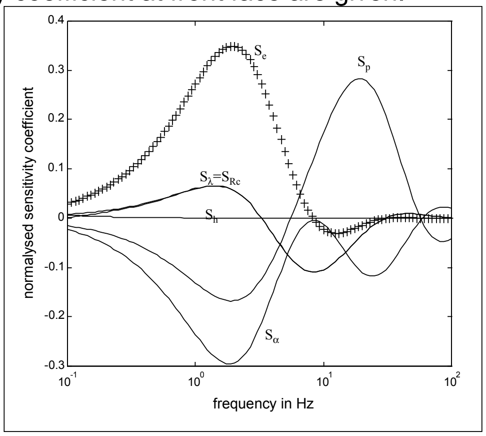

figure 5 : sensitivity coefficient at front face « standard gluing case »

The sensitivity coefficients for all parameters are null towards the low and high frequencies since the phaselag tends towards a constant value.
All sensitivity coefficient are of the same order of magnitude except for h which is null all over the area of calculation.
A local correlation appears between λ and R_c.
All the other curves seem to be uncorrelated two by two.

To conclude, ones must keep in mind that :
- front face measurements have same order and bounded sensitivity coefficients and are sensitive to p.
- rear face measurements have an ultra sensitivity to e and α, a null sensitivity to p.
- Rc and λ can be estimated only through the product $R_c\lambda$

OPTIMAL DESIGN FOR NDC STUDY OF GLUINGS

One is interested in estimating the highest number of parameters in one experiment. The frequencies area chosen is the largest reachable one with the « standard gluing » case (0.1Hz<f<20Hz). It is supposed to have 30 measurements points in this area. Thus the problem is the choice of front face or rear face or both configuration. The tool used is the condition number of matrix X'X and if there is a doubt the calculation of the inverse of ($1/\sigma^2$*X'X).
The matrix X'X is built with partial derivative versus normalized parameters to avoid bad condition number caused by different order of magnitude of these parameters.

A bad conditioning of matrix X'X can be due to :
- a lack of information on one or more parameters
- a local correlation between two or more parameters.

The method used is to consider the problem of the measurement of 1 parameter with the knowledge of the other 5, then 2 parameters with the knowledge of the other 4 , the last step being the estimation of 6 parameters at the same time. For each problem all the cases are considered.

Rear face configuration

1 parameter with the knowledge of the 5 others
This case demonstrates that rear experiments do not give information on h and p.
2 parameters with the knowledge of the 4 others
All the couples of properties can be estimated simultaneously excepted those includind h and p, excepted (λ,Rc) and (e,α) which present a high level of correlation.

Some studied cases and their conditioning are given.

Studied case	conditioning
(α,λ)	114.8
(α,R_c)	115
(α,e)	**1555**
(λ,R_c)	**42634**
(λ,e)	87
(R_c,e)	88

One musts notice that the couple (α,R_c) can be estimated, which is quite important for laboratory applications where p and e can be supposed to be known with accuracy.
3 parameters with the knowledge of the 3 others
no 3-uplets can be estimated.

Front face configuration

1 parameter with the knowledge of the 5 others
This case demonstrate, opposite to rear face ones, that front face measurements carry informations about p. There is always a lack of information about h.
2 parameters with the knowledge of the 4 others
All the couples of parameters can be estimated excepted those including h, and excepted (λ,R_c).
3 parameters with the knowledge of the 3 others
All the 3-uplets of parameters can be estimated excepted those including h, and (λ,R_c). The 3-uplets (α,R_c,p) can be estimated which gives a high performance of this periodic method to CND of the gluing.
4 parameters with the knowledge of the 2 others
No 4-uplets can be estimated.

Rear and front face configuration

Performing experiment on the two faces do not improve the conditioning of the estimations. The 2-uplets and 3-

uplets which can be estimated remain the same those for front face configuration. However, variance of the estimates are improved by taking the measurements from the two faces : front face measurements allows to estimate 3-uplets and rear face measurements carry great information on α.

OPTIMAL DESIGN FOR R_c MEASUREMENT

In this chapter, the aim is to measure R_c with high accuracy, the other parameters being supposed to be known. For front and rear face configuration, the optimal frequencies of study (lowest frequency, highest frequency) will be calculated. Influence of error on SBK parameters is study.

Optimal conditions minimize the variance of the estimate. The following development which calculates estimated variance, is dedicated to linear problem. However in most cases of non-linear problem under the assumption of gaussien white additive noise, least square estimator gives performance similar to those calculated given by linear assumption.

One supposes a linear problem with N measurements Y. These N measurements depend on p parameters θ be separated in two families of p_i unknown parameters to estimate and of p_c SBK parameters. The matrix of sensitivity is switched in two submatrix one for the SBK parameters, the other for unknown parameter $X=[X_i|X_c]$. ε is the vector of N noises of measurement (gaussian white zero mean value noise with constant variance σ^2).

For linear or non linear case, if G is a model of the experiment, X is a (N,p) matrix with $X(i,j)=\dfrac{\partial G(i)}{\partial \theta_j}$ where i is linked to a specific experimental setting. X' denotes X transpose.

For linear case :

$$Y=\begin{bmatrix} X_i & X_c \end{bmatrix}\begin{vmatrix} \theta_i \\ \theta_c \end{vmatrix}+\varepsilon \qquad /9/$$

So, for θ_i :

$$X_i\,\theta_i = Y - X_c\,\theta_c + \varepsilon \qquad /10/$$

Under the assumption made on measurement noise, the optimal estimator is the least square one and is given by :

$$\theta_{i_i}^{lsq}=(X_i^{'}X_i)^{-1}X_i^{t}(Y-X_c\theta_c) \qquad /11/$$

The error of measurement on SBK parameters is taken into account through random nature of vector θc normally distributed around its meaning true value.

If θ_c is unbiased, θ_i^{lsq} is unbiased (on average on the true parameters).

Variance of θ_i is calculated :

$$\mathrm{cov}(\theta_i^{lsq})=E((\theta_i^{lsq}-E(\theta_i^{lsq}))(\theta_i^{lsq}-E(\theta_i^{lsq}))^t) \qquad /12/$$

where E is the statistical average.

so

$$\mathrm{cov}(\theta_i^{lsq})=(X_i^{'}X_i)^{-1}X_i^{'}(Y-E(Y)-(X_c\theta_c-E(X_c\theta_c))) \times (Y-E(Y)-(X_c\theta_c-E(X_c\theta_c)))'X_i(X_i^{'}X_i)^{-1} \qquad /13/$$

The central term function of θ_c and Y can be written as :

$$E((Y-E(Y))((Y-E(Y))')=\mathrm{cov}(Y) \qquad /14/$$

$$E((X_c\theta_c-E(X_c\theta_c))(X_c\theta_c-E(X_c\theta_c))')=X_c\,\mathrm{cov}(\theta_c)X_c^{'} \qquad /15/$$

If the noise on Y and θ_c are independent :

$$E((X_c\theta_c-E(X_c\theta_c))(Y-E(Y))')=0 \qquad /16/$$

with the following assumptions :

$$\mathrm{cov}(Y)=\sigma_Y^2 I \text{ et } \mathrm{cov}(\theta_c)=\sigma_c^2 I\,, \qquad /17/$$

ones obtain :

$$\mathrm{cov}(\theta_i^{lsq})=(X_i^{'}X_i)^{-1}X_i^{'}(\sigma_Y^2 I+\sigma_c^2 X_c X_c^{'})X_i(X_i^{'}X_i)^{-1} \qquad /18/$$

Covariance of θ_i^{lsq} contains the $X_i^{'}X_i$ term which carry information on θ_i contained in Y, the σ_Y^2 term which expresses the power of the noise of measurement and the $\sigma_c^2 X_c^{'} X_c$ term which expresses the influence of the error made on θ_c on the covariance of θ_i. The variances of estimates increase with the noise of measurement with errors on SBK parameters, and decrease with information brought on the parameters to estimate.

One expresses this formula in the study of gluings where R_c has to be estimated and α is known with a variance of σ_α^2. With N measurements made at N frequencies f(i) :

$$\mathrm{var}(R_c)=\frac{1}{\sum_{i=1}^{N}\left(\frac{\partial\varphi(f(i))}{\partial R_c}\right)^2}\left(\sigma_\varphi^2+\sigma_\alpha^2\sum_{i=1}^{N}\left(\frac{\partial\varphi(f(i))}{\partial\alpha}\right)^2\right) \qquad /19/$$

The goal is to minimize var(R_c) versus the frequencies used.

If $\sigma_\alpha^2=0$, α is perfectly known, one has to maximize the term $\sum_{i=1}^{N}\left(\frac{\partial\varphi(f(i))}{\partial R_c}\right)^2$ which conducts to place all the points at the same frequency. This frequency maximizes the magnitude of sensitivity coefficient of phaselag versus R_c. The optimization is made for the « standard gluing » case.

Configuration	number points	σ_φ	optimal frequency	σ_{Rc}/R_c optimal
rear face	30	1°	8 Hz	0.010
front face	30	1°	8.37 Hz	0.029

If the diffusivity α is known with error, a compromise is necessary between information term on α and on R_c. A minimization on the lowest frequency (f_{min}) and on the largest frequency (f_{max}) is made with 30 points in the area. For any case, it is found that f_{min}=f_{max}. This is due to only one parameter is to be estimated then one has to concentrate all the measurements at the optimal frequency.

A study for the rear face configuration with 30 points and σ_φ=1° gives :

σ_α/α	optimal frequency	σ_{Rc}/Rc optimal
0%	8Hz	0.01
2%	3.07Hz	0.05
5%	1.98Hz	0.118

A study for the front face configuration with 30 points and σ_φ=1° gives :

σ_α/α	optimal frequency	σ_{Rc}/Rc optimal
0%	8.37Hz	0.029
2%	8.38Hz	0.029
10%	8.41Hz	0.029

For rear face configuration and for a null error on diffusivity, the optimal point is the maximum of sensitivity. But at this frequency, the sensitivity of diffusivity is high. (see figure 4). So as soon as an error is considered on α, the optimal frequency decreases. As consequences measurements are less sensitive to α but less sensitive to R_c. The optimal level of error increases.
For front face configuration, the maximum of sensitivity to R_c is reach for a null value of sensitivity to α (see figure 4). The optimal frequency and the optimal accuracy are insensitive to error on diffusivity.
Therefore this configuration can be considered as the best one although the sensitivity coefficient to R_c for front face is lower than for rear face (see case σ_α=0).
One musts remark that the best configuration is the one where the unknown parameters have at least the same order of sensitivity as the SBK parameters. A configuration where sensitivities to the unknown parameters are good but where there are ultra sensitive SBK parameters should be avoided.

To validate the use of periodic method for the measurement of R_c, accuracy of the estimated value is calculated with error on all the other parameters : 2% error on λ, 2% error on α, 10µm on e and p, 2% error on h, 1° on phaselag measurements.
Two cases are studied, 30 points on optimal frequency and 30 points over 0.1Hz < f < 20Hz.

configuration	optimal frequency	σ_{Rc}/R_c
rear face	2.94 Hz	0.057
front face	7 Hz	0.039

configuration	optimal frequency	σ_{Rc}/R_c
rear face	0.1Hz<f<20Hz	0.075
front face	0.1Hz<f<20Hz	0.105

This two tables demonstrate the adequation of the periodic method to measure Rc since accuracies inferior to 10% are reachable with 30 experimental points. A good choice of configuration and experimental points can divide error of estimation by a factor 2.

CONCLUSION

This communication describes a method to study gluings at millimeter scale. A modulated laser beam heats a gluing at its front face. The phaselag of thermal field is recorded at front or rear face versus the frequency of excitation. A model is developed with thermal quadrupoles. Parameters are estimated by fitting experimental and theoretical curves with least square estimator.

One musts have in mind that only the product Rc.λ is reachable.

A global method of optimal design of experiment is applied.

It is demonstrated that experiments at front face allow application to CND. The 3-uplets (α,p,R_c) can be estimate in one experiment with the assumption of the knowledge of λ,h,e.

ODE allows to find the optimal experimental condition with or without taken into account error of measurement on SBK parameters. Accuracy lower than 10% can be hoped.

First experiments will be carried out where the problems of 3D heat transfer and no punctual nature of the gluing will be considered.

ACKNOWLEDGEMENTS

I want to acknowledge Mr Sayous (DGA/DCE/CTA/LOT/GHF) for his technical support.

REFERENCES

[1] C.GERVAISE, C.NOUALS, M.CZERNIAK, C.CALDERAN, S.BENET, JJ.SERRA
"Propriétés thermiques des matériaux hétérogènes à l'échelle mésoscopique: simulations et méthodes d'inversion", Congres français de thermique, mai 1997, Toulouse, France

[2] C.GERVAISE, C.NOUALS, C.CALDERAN, B.CLAUDET, JJ.SERRA
"Multiscale diffusivity measurements by periodic methods" 24th International Thermal Conductivity Conference, octobre 1997, Pittsburgh, USA

[3] C.GERVAISE, C.NOUALS, C.CALDERAN, S.BENET,

JJ.SERRA
« Identification de propriétés thermophysiques par méthodes périodiques : approches multi-échelles »
Ecole d'hiver METTI (Métrologie Thermique et Techniques Inverses) 25-30 Janvier 1999 Odeillo France

[4] LEPOUTRE (F), BALAGEAS (D), FORGE (P), HIRSCHI (S), JOULAUD (J.L)
« Micron-scale thermal characterizations of interfaces parallel or perpendicular to the surface ». J.Appl.Phys, 1995, 78(4), p 2208-2233

[5] OZISIK (M.N)
Heat Conduction . second edition John Wiley&Sons, New York, 1993

[6] KAY (S.M)
Fundamentals of statistical signal processing, estiamtion theory PTR Prentice Hall, 1993,
ISBN 0-13-042268-1

[7] BECK (J.V), ARNOLD (K.J)
Parameter Estimation In Engineering and Science , John Wiley&sons

[8] RAYNAUD (M)
« Methods for experimental design and parameter identification », 14th European Conference on Thermophysical Properties, Lyon, France, 1996

[9] SODERSTROM, STOICA
System identification Prentice Hall, 1989, ISBN 0-13-881236-5

