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Photothermal infrared thermography applied to the identification of thin layer thermophysical properties

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Abstract:

The aim of the present work is the thermal non-destructive characterisation of layers at the surface of metals. The sample is sinusoidally heated by means of an argon ion laser and a focal plane array infrared camera (CEDIP IRC 320-4 LW) is used to measure the temperature variations at the surface of the layer. A numerical lock-in procedure allows the detection of very weak temperature variations at the surface of the sample, down to a few mK when working from the acquisition of hundreds of images, yielding amplitude and absolute phase maps for modulation frequencies ranging from 0.1 Hz to 1000 Hz.

An inverse procedure uses the Gauss-Newton parameter estimation method, in order to identify the thermal conductivity and the optical absorption coefficient of the layer. Confidence intervals on the parameters can also be estimated by the inverse procedure. More particular attention is devoted to the study of the sensitivity coefficients, as functions of the frequency range and of the radial range along the profiles, in order to optimise the identification procedure.

1. Introduction

Thin coating properties, and particularly their thermal conductivity, are not the same than the bulk values [1]. The interphase is usually defined as the part of the coating which has not the properties of the bulk [2]. Afterward, we suppose that the thickness of the investigated coating is smaller than the interphase thickness and that it is homogenous.

Infrared thermography allows to get a map of temperature at the surface of a sample. This gives some information on the heat diffusion in both directions: radially and in the depth. The different methods to measure thermal properties of materials are generally techniques which use only one infrared detector [3,4]. The recent development of focal plane array cameras brings an improvement of the thermal and spatial resolution with however a loss of the time resolution.

The method used in this work is a “front face” method in which the sample temperature response (amplitude and phase lag) due to a laser excitation is recorded. With a parametric identification procedure, we determine the thermal conductivity and/or the heat capacity and/or the optical absorption coefficient.

2. Experimental set-up

The camera used here is an infrared focal plane array camera CEDIP IRC 320-4 LW. The sample is heated with an argon-ion laser beam (488 and 514.5 nm) which is sinusoidally modulated using an acousto-optical modulator. The experimental set-up (fig. 1) allows a “front face” excitation with normal incidence. The infrared response is recorded by the camera in a spectral window ranging from 7.5 to 10 µm. A numerical lock-in procedure gives the amplitude and phase lag maps of the harmonic response at the surface of the sample. The signal to noise ratio is improved by the accumulation of several hundred of pictures.

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3. Thermal modelling: direct model

The first step is the resolution of the heat transfer equation:

$$\Delta T(\vec{r},t) - \frac{1}{a}\frac{\partial T(\vec{r},t)}{\partial t} = -\frac{q(\vec{r},t)}{k} \tag{1}$$

where $q(\vec{r},t)$ is the heat source, a the thermal diffusivity and k the thermal conductivity.

In our study, the excitation is sinusoidally modulated with the pulsation ω. This implies that the temperature response $T(\vec{r},t)$ is also sinusoidal and can therefore be written:

$$q(\vec{r},t) = q(\vec{r}).e^{j\omega t} \qquad\qquad T(\vec{r},t) = T(\vec{r}).e^{j\omega t} \qquad \text{(2) and (3)}$$

where $T(\vec{r})$ is complex due to the phase lag with the heating source.

So the heat equation becomes:

$$\Delta T(\vec{r}) - \frac{j\omega}{a}T(\vec{r}) = -\frac{q(\vec{r})}{k} \tag{4}$$

The laser excitation has bidimensional axisymmetric geometry which implies:

$$\frac{\partial^2 T(r,z)}{\partial r^2} + \frac{1}{r}\frac{\partial T(r,z)}{\partial r} + \frac{\partial^2 T(r,z)}{\partial z^2} - \frac{j\omega}{a}T(r,z) = -\frac{q(r,z)}{k} \tag{5}$$

To solve this equation, we combine an integral transform on the radial component r with a harmonic response [5]. A Hankel transform of zero order on the radial component is applied to the equation (4):

$$\frac{\partial^2 \overline{T}(\delta,z)}{\partial z^2} - \left(\delta^2 + \frac{j\omega}{a}\right)\overline{T}(\delta,z) = -\frac{\overline{q}(\delta,z)}{k} \tag{6}$$

where $\overline{T}(\delta,z)$ and $\overline{q}(\delta,z)$ are respectively the Hankel transform of the temperature and of the heat source and δ is the Hankel variable:

$$\overline{T}(\delta,z) = \int_0^\infty T(r,z)J_0(\delta r)r\,dr \qquad\qquad \overline{q}(\delta,z) = \int_0^\infty q(r,z)J_0(\delta r)r\,dr \qquad \text{(7) and (8)}$$

where J_0 is the Bessel function at zero order.

If the transform of the heat source $\overline{q}(\delta,z)$ is replaced in the expression (6) by a Dirac distribution in the axial component z (the symmetry of the problem allows us to take a plane excitation at the z' depth), we then obtain the equation that must verify the unidimensional harmonic answer $H(\delta,z|z')$:

$$\frac{d^2 H(\delta,z|z')}{dz^2} - (\delta^2 + \frac{j\omega}{a})H(\delta,z|z') = -\frac{\delta(z-z')}{k} \tag{9}$$

This equation is solved by searching the solution as the sum of the homogeneous equation (without excitation) and of the particular solution.

Integrating on z', the Hankel transform of the temperature is obtained:

$$\overline{T}(\delta,z) = \int_0^\infty H(\delta,z|z')\overline{q}(\delta,z')dz' \tag{10}$$

and finally, by the inverse transform, the temperature of the sample.

$$T(r,z) = \int_0^\infty\int_0^\infty H(\delta,z|z')\overline{q}(\delta,z')J_0(\delta r)\delta\,d\delta\,dz' \tag{11}$$

<u>Application to the bi-layer sample:</u>

In the case of a bi-layer sample, the interface (z=l) is modelled by an interfacial thermal resistor R_t without thickness. The boundary conditions on the temperatures are (the first index represents the coating and the index 2 represents the substrate):

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- exchange coefficient h at the interfaces (z=0 and z=d):

$$k_1 \left.\frac{\partial T_1(r,z)}{\partial z}\right|_{z=0} = h.T_1(r,z)\big|_{z=0} \tag{12}$$

$$-k_2 \left.\frac{\partial T_2(r,z)}{\partial z}\right|_{z=d} = h.T_2(r,z)\big|_{z=d} \tag{13}$$

- continuity of the flux at the interface (z=l):

$$-k_1 \left.\frac{\partial T_1(r,z)}{\partial z}\right|_{z=l} = -k_2 \left.\frac{\partial T_2(r,z)}{\partial z}\right|_{z=l} \tag{14}$$

- interface thermal resistor R_t at z=l:

$$T_1(r,z)\big|_{z=l} - T_2(r,z)\big|_{z=l} = -k_2.Rt \left.\frac{\partial T_2(r,z)}{\partial z}\right|_{z=l} \tag{15}$$

We search for the punctual harmonic response due to the plane heat source at the depth z'. H_{11} and H_{12} are respectively the response of the coating and of the substrate corresponding to a heat source in the coating. When the heat source is in the substrate, the resulting responses are H_{21} and H_{22}. The expression temperature across the layer is then:

$$T_1(r,z) = \int_{-\infty}^{+\infty} \left\{ \int_0^l H_{11}(\delta, z|z')\bar{q}_1(\delta,z')dz' + \int_l^d H_{21}(\delta, z|z')\bar{q}_2(\delta,z')dz' \right\} J_0(\delta r)\delta d\delta \tag{16}$$

The integration on z' is analytical while the inverse Hankel transform is numerical. The resulting temperature is a complex number.

4. Inverse problem, study of sensitivities

The camera does not give directly absolute temperatures but numerical levels proportional to the luminance of the sample. The surface temperature of the sample is composed of three terms (figure 2):

$$T = T_{amb} + T_{cont} + T_1(r,0) \tag{17}$$

where T_{amb} is the ambient temperature and T_{cont} is the continuous temperature.

As a first approximation (for small values of T_1(r,0)) [4,6], the radiation broadcast is proportional to the alternative temperature. However, to access to the absolute temperature (in Kelvin), some parameters must be known such as the emissivity of the sample, the power of the laser beam, the standard constants of the camera and also the optical properties of the sample like reflection coefficient. In order to eliminate these last parameters in the identification, we realise a normalisation of the signal at the working frequency with a signal at another frequency. The quantities used in the parametric identification are:

- for the theoretical data, a ratio of alternative temperatures
- and for the experimental data, a ratio of the luminances given by the camera.

These variables are proportional, represent the same phenomena and thus are comparable. The working frequency is chosen in order to have a thermal diffusion length approaching the thickness of the coating. To choose the normalisation frequency, the sensitivity of the normalised value versus the normalisation frequency for a working frequency of 10.5 Hertz is plotted in figure 3. It can be seen that a high frequency is better to normalise the radial profile in manner to have greatest sensitivities. In order to have variables with the same dimension and to use all the available information, the parametric identification is achieved with both real and imaginary parts of the complex temperature rather than with amplitudes and phase lags.

The surface temperature $T_1(r,0)$ depends of several parameters which have more or less influence. The parametric identification depends of the sensitivity of the model at these different parameters. If it is sensible to a parameter, this parameter is easily identifiable. The parametric estimation method used in this work is a Gauss-Newton algorithm which corrects

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the parametric vector $m_{(n)}$ at each iteration n:

$$\delta m_{(n)} = -\left(G^t_{(n)} G_{(n)}\right)^{-1} G^t_{(n)} \left(d_{th}(m_{(n)}) - d_{exp}\right) \tag{18}$$

where d_{th} and d_{exp} are respectively the theoretical and experimental data. This correction depends of the sensitivity matrix G_n. If two parameters present sensitivities which are proportional, the identification algorithm can not converge because the sensitivity matrix has some collinear vectors: the parameters are said to be correlated.

The above algorithm also allows to determine the different uncertainties Δm^r caused by the bad knowledge of parameters such as specific heat and thickness of the coating, thermal conductivity and specific heat of the substrate and the radius of the laser beam. These uncertainties depend on the different sensitivities $G_{(n)}$ and on the uncertainties on these parameters $G^c_{(n)}$ (these uncertainties are estimate at the end of the identification, for the latest index n):

$$\Delta m^r_{(n)} = \left(\left(G_{(n)}\right)^t G_{(n)}\right)^{-1} \left(G_{(n)}\right)^t \left[G^c_{(n)} \Delta m^c\right] \tag{19}$$

More sensitive the parameter is, more precisely the parameter must be known. The different results of these uncertainties are resumed in the table 1.

5. Experimental results

The infrared camera CEDIP IRC 320-4 LW allows acquisition of images of 320x240 pixels of 30x30 micrometers. The image acquisition frequency and their dimensions can be modified. Figure 4 shows the radial profile of amplitude and phase lag of the complex temperature given by the numerical lock-in procedure. The modulation frequency used here is 10.5 hertz. The investigated sample is composed of an epoxy resin layer with a thickness of 40 micrometers onto an aluminium substrate with a thickness of 1.5 millimeters. The thermal resistance is neglected [7]. The radius of the laser beam, measured with a beam analyser, is 0.7 millimeter. After the numerisation by the acquisition system, the amplitude is represented by numerical levels (NL). The amplitude value of normalisation is obtained in the same experimental conditions and at a frequency such as the thermal regime is different, *ie* 2000 Hz. This allows to undertake the identification with all the experimental data: real and imaginary parts.

The parametric identification yields two parameters: the thermal conductivity and the optical absorption coefficient of the coating. These two parameters are not well known: the thermal conductivity because it is not the same than the bulk value and the optical absorption coefficient because it is introduced by the experiment.

The starting values are 0.2 $W.m^{-1}.K^{-1}$ for the conductivity and 10^5 m^{-1} for the optical absorption coefficient. The identification procedure converges to final values of 0.132 $W.m^{-1}.K^{-1}$ and 211400 m^{-1}. The corresponding experimental and theoretical radial profiles are compared on figure 5. The correlation of the residues shows however that the model does not well describe the experiment or that some parameters considered as well known are not sufficiently correct. Table 1 presents the different uncertainties generated by the identification and by the uncertainty on the parameters supposed to be known.

6. Conclusion

Photothermal infrared radiography using a focal plane array camera allows to identify two parameters of a thin layer of epoxy resin onto an aluminium substrate. using of only one radial profile gives enough information for the parametric algorithm to converge. An improvement of the time resolution of the camera could bring more sensitivity and could allows the identification of three or more parameters. The uncertainty results show that the thickness of the coating is an important parameter and that its value must be precisely determined.

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	Ident.	Cp_1	k_2	Cp_2	l	r_0
Δk_1	4,2	5,2	0,2	0	10,2	1.4
$\Delta\beta_1$	5,8	0,2	0,2	0	5,2	15.9

Table 1 : relative uncertainties (in %) on the identified parameters du to the identification and du to the bad knowledge of some parameters.

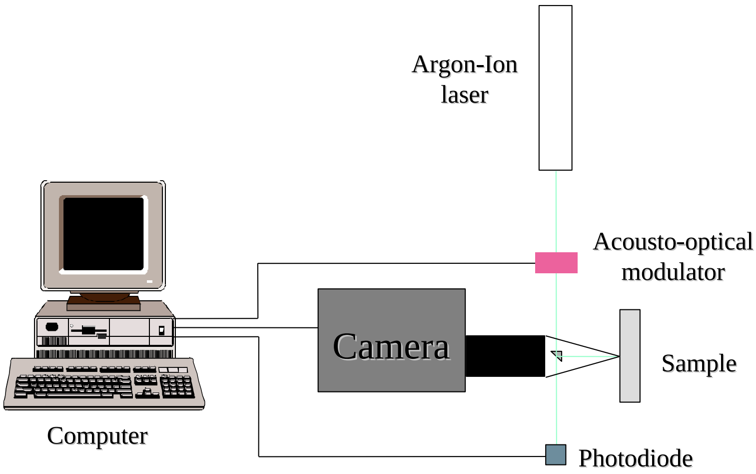

Figure 1 : experimental set-up

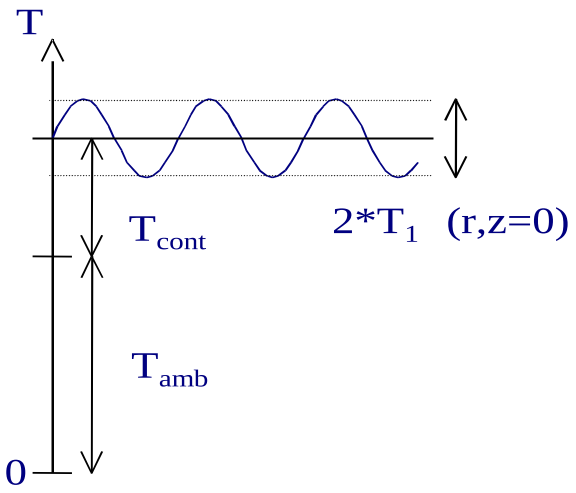

Figure 2 : temperature definitions

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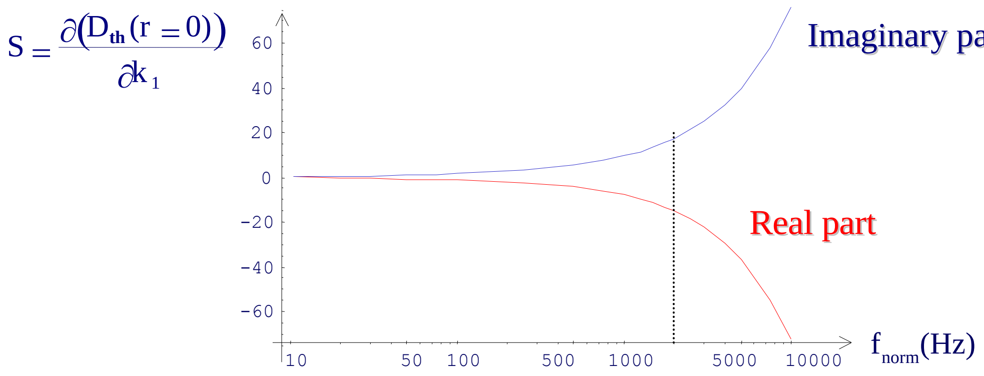

Figure 3 : Sensitivity of normalised value versus the normalised frequency

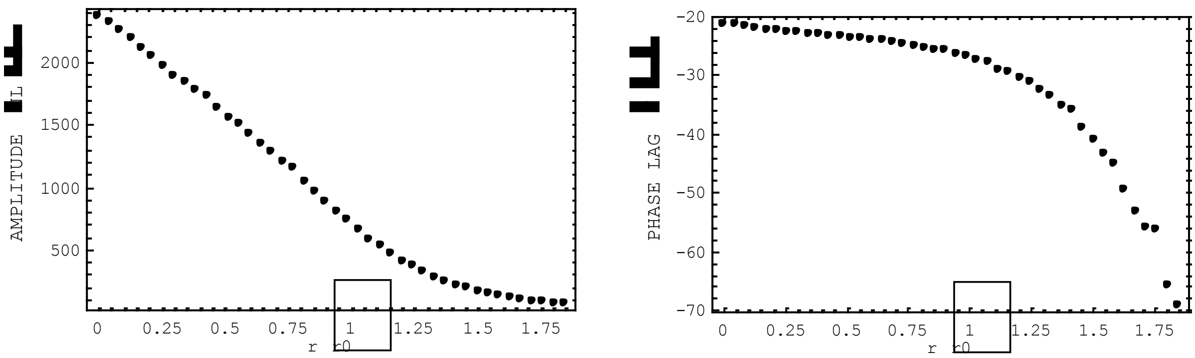

Figure 4 : amplitude and phase lag profiles

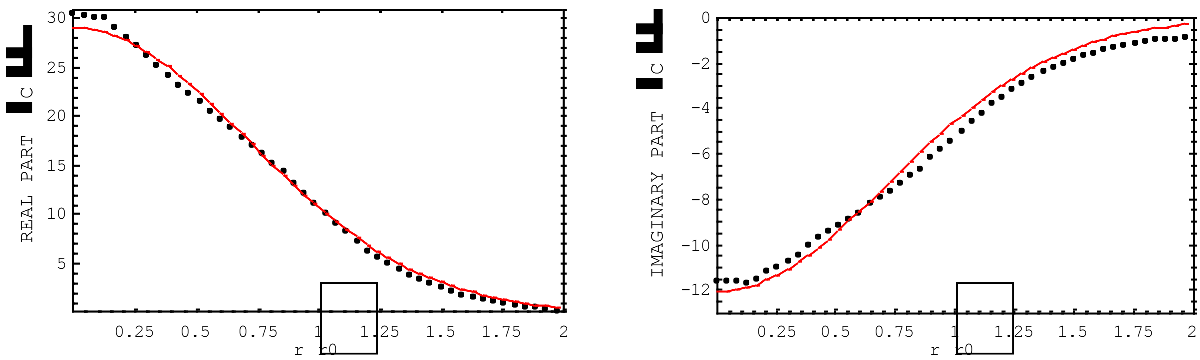

Figure 5 : experimental (dotted line) and theoretical (full line) profiles of real and imaginary parts at the end of the identification