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Analysis of boundary conditions and transient signal treatment in diffusivity measurements by laser pulse method

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Résumé. — Une méthode de détermination de la diffusivité par méthode flash-laser est analysée. Une technique de mesure permettant d'évaluer la distribution d'énergie et la forme de l'impulsion laser est développée. Cette technique qui peut être facilement implantée sur chaque expérience de mesure de diffusivité par une méthode flash permet de contrôler périodiquement les caractéristiques des flashes, caractéristiques dont l'importance est primordiale pour la précision des mesures de diffusivité. On discute également des corrections du modèle classique de Parker *et al.* [1] pour ce qui concerne les pertes de chaleur et la durée non infiniment petite de l'impulsion. L'amélioration de la précision des mesures de diffusivité en utilisant un système d'acquisition de données digitalisé est proposée. Une technique de lissage du signal expérimental transitoire est développée en s'appuyant sur une méthode de transformée numérique de Fourier. Finalement on présente le dispositif de mesure de diffusivité ainsi que quelques résultats obtenus pour des matériaux standards.

Abstract. — A laser pulse method for diffusivity determination is analysed. The method which permits the measurement of the laser pulse energy distribution as well as its shape is developed. This method can be easily implanted into laser pulse diffusivity set-up and it allows to check periodically the pulse characteristics that is of primary importance for the accuracy of the diffusivity measurements. The corrections to the classical model of Parker *et al.* [1] due to the heat losses and finite pulse duration are then discussed. The improvement of the accuracy of the diffusivity measurement through the use of a digitalized data acquisition system (DDAS) is proposed. The smoothing technique of the experimental, transient thermal signal is developed on the basis of a digitalized Fourier transform. Finally the diffusivity set-up is presented and some results for the standard materials are shown.

1. Introduction.

The rapid implementation of small digital systems in field of the thermal properties measurements makes it necessary to adapt the classical methods to the numerical tools particularities. The flash method widely used to determine the thermal diffusivity, based on the paper of Parker *et al.* [1], is one of the best known and seems to be a very good example

for such an adaptation. This method has been reviewed many times e.g. by Taylor [2] and [3] or Righini and Cezairliyan [4].

Although several authors as e.g. Perovič and Maglič [5] or Koski [6] have already discussed the advantages of the use the digitalized data acquisition system (DDAS) for the diffusivity measurements; problems such as the analysis of the boundary conditions, which should be controlled systematically when one uses laser pulses and/or numerical filtering, however discussed by Koski [6], needs still more attention. In the present analysis we would like to emphasize a method for the periodic control of the laser pulse characteristics (such as pulse energy distribution, duration and

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shape), characteristics very important because they are the correction base of the mathematical model when the effects of finite pulse duration are important.

As in many cases this effect can be dissociate from the heat losses correction this one will be discussed separately.

2. Analysis of the boundary conditions.

2.1 ENERGY AND SHAPE OF THE LASER PULSE. — The knowledge of the energy (and its spatial distribution) of a laser pulse striking the sample during a diffusivity measurement is important for the two following main reasons :

1° The value of the energy permits to estimate the maximal increase of the rear face sample temperature T_{\max} :

$$T_{\max} = \frac{Q}{Sc_p \rho L} \quad (1)$$

where Q is the pulse energy, S is the sample surface area, L its thickness and ρ , c_p are respectively its density and specific heat. Moreover, in some cases as e.g. for measurements near the temperature of a material phase transformation it is desirable to know with precision the temperature excursion (T_{\max}) resulting from the flash.

2° The inhomogenous spatial distribution of the laser energy can lead to errors in the diffusivity determination when the method of Parker *et al.* [1] is used. As shown by Beedham and Dalrymple [7] the energy profile of the laser pulse with a minimum pronounced at the centre results in errors in diffusivity determination up to 8 %, depending on the focusing of the detector monitoring the rear face temperature.

The system we have developed to control the energy distribution of the laser beam is shown in figure 1a. The Nd : glass laser (1) pulse strikes the extremity of the high attenuation optical fibre (3) which diameter is 0.25 mm. The attenuated light is detected by a rapid photodiode (type PIN) circuit (2) and the resulting voltage variations are registered by a digital storage oscilloscope before being introduced into the memory of a microcomputer. The fiber is moved normally to the optical axis. A typical evolution the signal with time, registered by the photodiode at a given point of the laser pulse, is given in figure 2. The integral *versus* time of such a signal is proportional to the energy of the pulse received by the optical fibre at a given point. As the pulse has circular symmetry it is sufficient, in order to determine the energy spatial distribution, to move the fibre only in one direction (passing through the centre of pulse and orthogonal to the optical axis). Such energy distributions for the laser charged respectively under 1.30, 1.65 and 2.0 kV are shown in the figure 3 and the pulse duration transformation is given in figure 4. The energy of the pulse can be more than 50 % higher in the outer

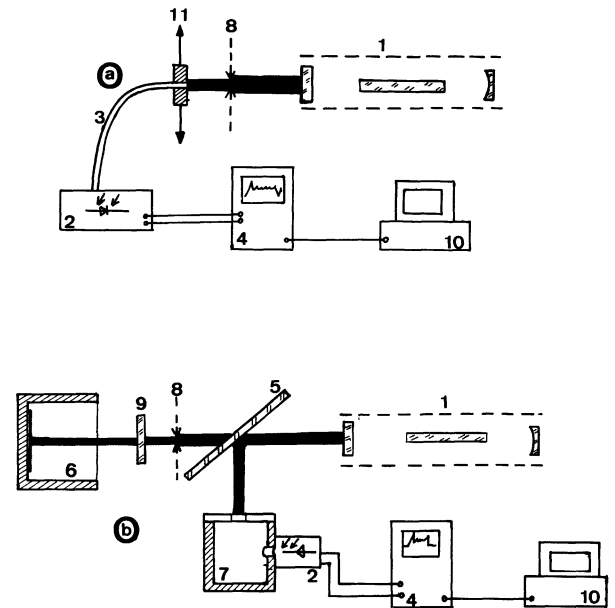


Fig. 1. — Sketch of the experimental arrangement for the determinations : a) of the laser pulse energy distribution and shape. b) of the laser energy. 1) Laser, 2) fast photodiode circuit, 3) optical fibre, 4) digital storage oscilloscope, 5) beam splitter, 6) commercial laser powermeter, 7) home made powermeter, 8) diaphragm, 9) silica window, 10) microcomputer, 11) mechanical device to move the optical fiber orthogonally to the optical axis.

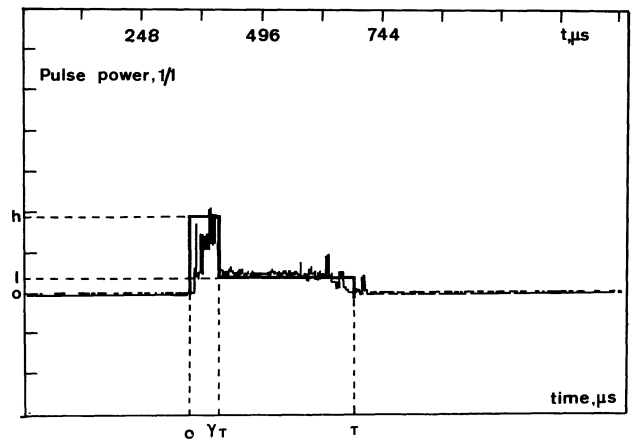


Fig. 2. — Typical temporal evolution of the laser pulse power (laser charged under 1.65 kV).

part of the pulse that in its centre (see Fig. 3a). For the same conditions the distribution of the pulse duration changes also very sharply. As one can deduce from the figures 3 and 4 the homogeneity of the pulse energy and duration distributions is better when the energy input to the laser is higher.

A careful analysis of figures 3a and 4a permits to find a shift of the pulse centre of about 1 mm from the optical axis. Thus the described above method has the advantage to give the possibility to achieve a good alignment of the optical axis.

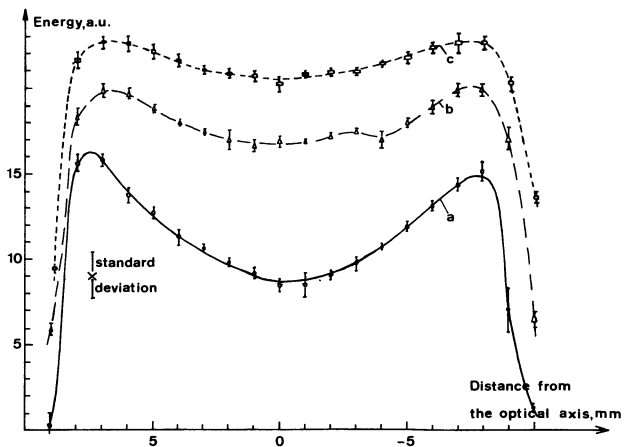


Fig. 3. — Spatial distribution of the pulse energy when the laser is charged respectively under 1.3 kV-a, 1.65 kV-b and 2 kV-c. The distance from the laser cavity is of about 1.5 m.

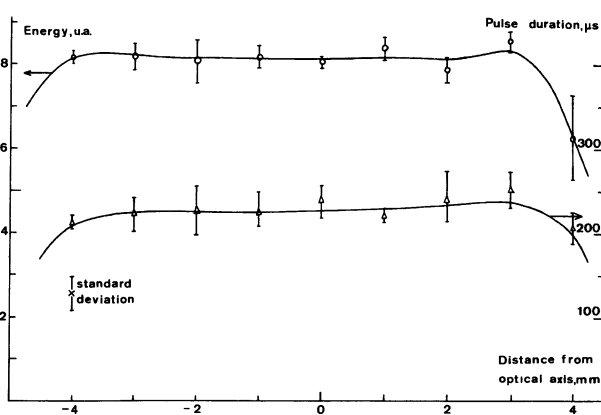


Fig. 5. — Spatial distribution of the pulse energy and duration when the laser is charged under 1.3 kV. The distance from the laser cavity is of about 1 m and a diaphragm ($\varnothing = 8$ mm) is used.

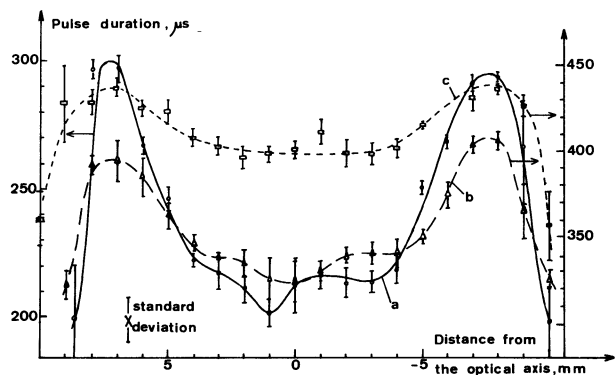


Fig. 4. — Spatial distribution of the laser pulse duration when the laser is charged respectively under 1.3 kV-a, 1.65 kV-b and 2 kV-c. The distance from the laser cavity is of about 1.5 m.

The use of a diaphragm having an internal diameter equal to that of the analysed sample i.e. 8 mm allows to obtain a better pulse spatial homogeneity and to reduce the overall pulse energy. This is confirmed by the results of the measurement made with such a diaphragm as shown in figure 5.

The appropriate shape of the pulse can be determined using a photodiode but an extreme care has to be taken in the choice of the time constant of the photodiode circuit. When one uses a time constant of about 10^{-8} s the shape of the pulse is as shown in figure 2 while when this constant is equal to 10^{-5} s the temporal form of the pulse is similar to a triangle. Knowing that the laser light waves have frequencies higher than 10^{13} Hz one can state that the energy coupling between laser pulse and solid is realized exclusively by the electrons. This means that heating of solid is done by the electrons too. That is why the temporal form of the light pulse has to be determined as much precisely as possible i.e. when measuring with a photodiode, by keeping the time constant as low as possible.

It is, for example, possible that Taylor and Clark [8] who used a method similar to ours but with a time constant of about 4×10^{-5} s might have a real laser pulse temporal shape different from the one they have shown.

The overall pulse energy is measured using a calorimeter (Sciantech model 360 401). As shown in figure 1b the laser pulse is transmitted through a silica window (9) as during diffusivity measurements. The results of the energy determination with precision within 10 % are presented in figure 6. Knowing the total energy of the pulse one can easily calibrate the sample calorimeter ((7) in Fig. 1b) which receives, thanks to a beam splitter (5), a constant fraction of the pulse energy. This calibration allows to determine the energy of the pulse during diffusivity measurements.

2.2 ABSORPTION OF THE ENERGY PULSE BY THE SAMPLES. — If the energy of the pulse is distributed uniformly onto the sample's surface as shown in figure 5,

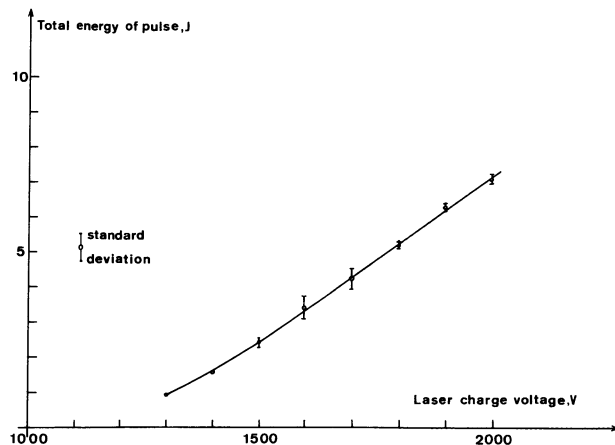


Fig. 6. — Energy received by the front face of the sample vs. laser charge voltage.

one can express the density of the thermal power P_1 as :

$$P_1 = \frac{Q}{\tau \cdot S}, \quad (2)$$

where τ is the pulse duration and Q , S have been defined in (1). As the thermal power is partially reflected by the surface the absorbed power density P_a at a distance x from the sample front face is equal to :

$$P_a = (1 - R) \cdot P_1 \cdot \exp(-\beta x), \quad (3)$$

where β is the optical absorption coefficient and R is the reflection coefficient. Both ones are related to the complex refractive index $n^* = n + ik$ by the well known expressions (see e.g. [9]) valid for the normal incidence :

$$R = \frac{(n - 1)^2 + k^2}{(n + 1)^2 + k^2} \quad \text{and} \quad \beta = \frac{4 \pi k}{n \lambda_0} \quad (4)$$

where n is the refraction index, k the extinction coefficient and λ_0 the thermal pulse wavelength in vacuum. For the typical wavelengths used in diffusivity measurements (red or near infrared light) the metals have a very high reflectivity $R \approx 0.6-0.9$ or even higher. But it is relatively easy to decrease the reflectivity by a simple treatment of the sample surface as a sand-blasting, black painting or both ones as we did for the measurements of the Armco iron diffusivity (see § 5).

Taking, for the sake of simplicity, that $R = 0$ it is possible to estimate (Table I) the depth of the optical absorption ⁽¹⁾ for various metals and oxides.

On the other hand the depth of the heat diffusion ⁽²⁾ may be expressed as $\sqrt{4 a \tau}$, where a is the thermal diffusivity and τ has been previously defined. The approximation of instantaneous pulse, used in the Parker *et al.* [1] model, corresponds to the condition :

$$\frac{1}{\beta} \ll \sqrt{4 a \tau}. \quad (5)$$

As shown in table I, for the considered metals this condition is easily fulfilled. However the dielectrics such as a metal oxides crystals are in most cases transparent in the visible and near infrared spectrum. The polycrystalline materials, as e.g. plasma sprayed ones, absorb better the light as one can observe in table II. For a typical technical ceramic as plasma sprayed zirconia, the transmittance and therefore absorptance depend highly on the material characteristics as microstructure, porosity etc... which are

⁽¹⁾ Defined as the distance from the sample irradiated surface where the power density has decreased by a factor of 1/2.718...

⁽²⁾ Defined as the distance from the surface of the irradiated sample where the excursion of temperature has decreased by a factor 1/2.718...

Table I. — Comparison between optical absorption depth and heat penetration depth for several metals and oxides at 300 K for an energy pulse duration $\tau = 500 \mu\text{s}$.

Material	λ_0 μm	n	k	a cm^2/s	β^{-1} μm	$\sqrt{4 a \tau}$ μm	Bibliography
Metals							
Fe	1.00	2.30	4.52	0.206	0.0405	203	Optical Properties[10] Diffusivity[11]
Ni	1.03	2.85	5.10	0.229	0.0458	214	
W	1.00	3.00	3.50	0.662	0.0682	364	
Ti	1.09	3.50	4.02	0.0925	0.0765	136	
Oxides							
Al ₂ O ₃				0.08		126	[11]
ZrO ₂ - 7.5 wt. % CaO				0.005		31.6	[12]

Table II. — Optical transmittance of plasma sprayed zirconia ceramics.

Material	Wavelength μm	Thickness μm	Transparency %	Bibliography
ZrO ₂ - 12 wt. % Y ₂ O ₃	1 - 3	50	82	[13]
ZrO ₂ - 8 wt % CaO	1.06	450	14	[14]
		1 440	6	
ZrO ₂ - 8 wt % Y ₂ O ₃	1 - 3	140	< 5	[12]

dependent on the technology of fabrication. In most cases measurement of the diffusivity of ceramics requires the application of a thin metallic or graphite coating onto the front face of the sample to ensure the absorption of heat, to fulfil the condition (5) and finally to protect the infrared detector against the high energy laser pulse.

3. The transient signal treatment.

The evolution of the temperature of the rear face of the sample resulting from an instantaneous pulse is expressed by [1] :

$$T(L, t) = T_{\max} \left(1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(-\frac{n^2 a \pi^2 t}{L^2}\right) \right) \quad (6)$$

where T is the temperature, T_{\max} and L have been previously defined, t is time and a the thermal diffusivity. The equation (6) is valid when the sample does not exchange heat with the surroundings. The thermal diffusivity is usually determined at the point of this evolution corresponding to $T(L, t) = T_{\max}/2$. If the time coordinate of this point is $t_{0.5}$ the diffusivity is given by

$$a = \frac{1.38 L^2}{\pi^2 t_{0.5}} \quad (7)$$

The calculated evolution of the temperature for a sample having thickness $L = 0.0945$ cm and diffusivity $a = 0.0048$ cm²/s is shown in figure 7a. This signal was transformed to its Digital Fourier Transform (DFT) taking $N = 100$ points f_n up to the time $t = 5 t_{0.5}$ by the use of the following formula e.g. [16] :

$$\text{DFT}_m = \sum_{n=0}^{N-1} f_n \exp\left(\frac{2 \pi i m n}{N}\right); \quad m = 0, 1, \dots, N-1 \quad (8)$$

where the frequency corresponding to the point m is equal to $\nu_m = m/N \cdot T_s$, T_s being the sampling frequency (in the present case $T_s = 5 t_{0.5}/100$). The DFT vs. frequency curve is shown in figure 7b, and it is clear that its slope decreases monotonically with the increase of frequency. This slope reaches zero for the frequencies in the range between $\nu_{0.1}$ and $\nu_{0.01}$ (see Fig. 7b) corresponding to maximal DFT value 0.1 and 0.01 fractions.

The transient signal obtained experimentally through the measurement of the evolution of the rear face temperature of zirconia sample (uniformly heated at 1 320 K in the furnace) is shown in figure 8a. The signal is registered directly from the infrared temperature detector in order to avoid any deformation during amplification. The DFT curve of this signal, shown in figure 8b, has maxima at the following frequencies : 50, 100, 150 and 200 Hz, maxima due to

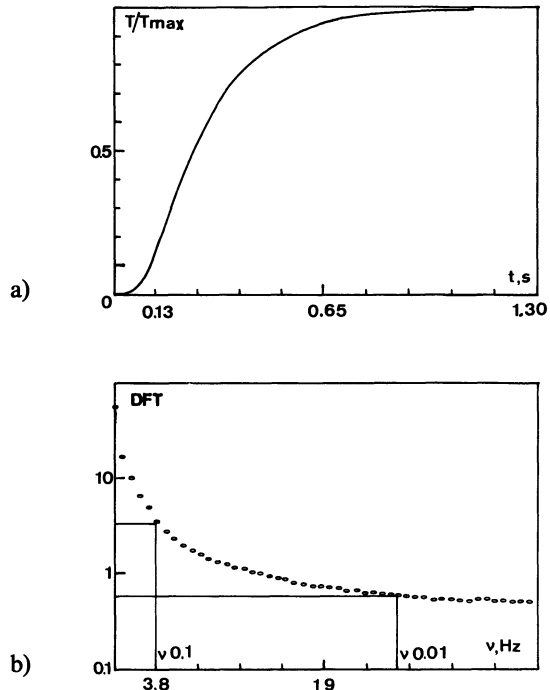


Fig. 7. — Theoretical transient signal corresponding to the evolution of the reduced temperature of the rear face of the sample calculated for $L = 0.0945$ cm and $a = 0.0048$ cm²/s — a) and its digital Fourier transform (DFT) calculated for the sampling period $T_s = \frac{5 \cdot t_{0.5}}{100}$ — b).

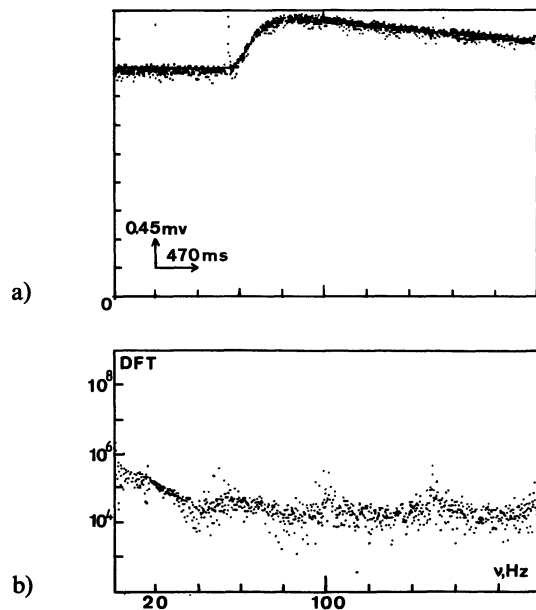


Fig. 8. — Experimental transient signal corresponding to the evolution of the temperature measured at the rear face of a zirconia sample having thickness $L = 0.0945$ cm at the temperature $T = 1 308$ K such as given directly by the infrared detector — a), and its DFT calculated for the sampling period $T_s = 2.5$ ms [12] — b).

the perturbation of the detector circuit by external electromagnetic fields (as discharge of the laser, furnace current supply etc...). The other possible fluctuations are mainly due to the photon noise of the detector.

Obviously, such a transient signal contains most informations in the low frequencies i.e. an experimental signal smoothing should be done, if one decides to use filters, by low-pass band filters. To achieve a better fitting of the filter characteristics it was decided to use the digital filters procedure (see e.g. [15, 16]).

There are generally two digital filtering procedures, recursive (IIR) filters and non-recursive (FIR) ones. Their direct comparison is difficult and depends on the application [16]. To simplify, one can state that the IIR procedures are more efficient in achieving given specifications of the filter (gain, ripple in pass-and stopband) than FIR procedures but FIR has no group delay distortion, having a linear variation of the phase *versus* frequency. Koski [6] has used the FIR filter to reduce the noise of the transient signal, being attracted by FIR filter zero phase shift property. We have tried to develop the IIR recursive procedure because of the more precise design of the filter gain *vs.* frequency, taking however into account the phase-shift of the filter.

The digital filter is assumed to be a linear weighting of the input data values. If the values of signal at the filter input are f_m and at its output — y_m , the recursive filter algorithm is given by the following relationship :

$$y_m = \sum_{n=-N}^N b_n f_{m-n} - \sum_{n=1}^N a_n y_{m-n} \quad (9)$$

where a_n and b_n are the weighting coefficients. In such a filter the output data depends on input ones as well as on previously computed output values.

The Butterworth filter procedure, we use, is said to be maximally flat near $\nu = 0$ and $\nu = \infty$ [15]. The power gain $|H_B|^2$ of such analog filter is equal to :

$$|H_B|^2 = \frac{1}{1 + \varepsilon^2 \left(\frac{\omega}{\omega_c}\right)^{2N}} \quad (10)$$

where $\omega_c = 2\pi\nu_c$, ν_c being the cut-off frequency (for all the frequencies below ν_c the power gain is higher than $\frac{1}{1 + \varepsilon^2}$), N is the filter order, found from the

inequality $N \geq \frac{\log\left(\frac{\lambda}{\varepsilon}\right)}{\log\left(\frac{\omega_r}{\omega_c}\right)}$ in which $\omega_r = 2\pi\nu_r$, ν_r being

the rejection frequency (for all the frequencies higher than ν_r the power gain is lower than $\frac{1}{1 + \lambda^2}$).

The above analog construction has been transformed *via* bilinear transformation [15] into a digital filter

leading to the determination of the weighting coefficients a_n and b_n of equation (9).

The following filter parameters should be determined : ε , λ , ν_r and ν_c and the resulting thereof N . Usually one takes $\varepsilon = 1$ and $\lambda = 3$. Tests have been made to find the best method to determine ν_c and ν_r . These tests were made using experimental signal nearly without noise obtained with a sample of POCO graphite ($L = 0.1$ cm) at 1 090 K. The influence of the choice of ν_c and ν_r onto the known diffusivity value of this sample was observed. The test conclusions showed that ν_c should be between $\nu_{0.1}$ and $\nu_{0.01}$ of theoretical DFT curve (constructed by taking the approximative value of sample diffusivity and exact value of its thickness — see Fig. 7b). In such conditions the heating part of the temperature evolution is transmitted through the filter without any change. The cooling part of this evolution is nearly not influenced by the filtering.

The recursive filter introduces a phase shift which depends on the signal frequency. As it is important, for the $t_{0.5}$ determination, to know exactly the beginning of the pulse — the group delay, i.e. the delay of the filter *vs.* frequency was determined (the details of such determination are given e.g. by Rabiner and Gold [16]).

Figure 9 shows the filter characteristics i.e. power gain and time delay *versus* frequency. The filter is adapted to the experimental signal shown in figure 8a (the filter parameters : $\nu_c = 7$ Hz, $\nu_r = 15$ Hz, $\lambda = 3$, $\varepsilon = 1$ and $N = 2$). The smoothing of the signal given in figure 8a by this filter is shown in figure 10a. The diffusivity is calculated under the assumption that the signal is shifted in time by the group delay mean value (computed up to rejection frequency — as shown in Fig. 9) and in the shown example is equal to 0.00476 cm²/s. The DFT of the smoothed signal (Fig. 10b) reveals the elimination of all the perturbations.

As it has been just discussed above that the smoothing procedure is applicable only in the case where the noise frequency spectrum is higher than the signal one.

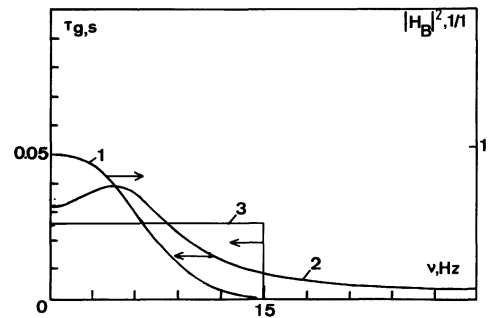


Fig. 9. — Evolution with the frequency on the : power gain (1), group delay (2) and mean group delay (3) for the recursive filter having the following parameters $\nu_c = 7$ Hz, $\nu_r = 15$ Hz, $\lambda = 3$, $\varepsilon = 1$ and $N = 2$.

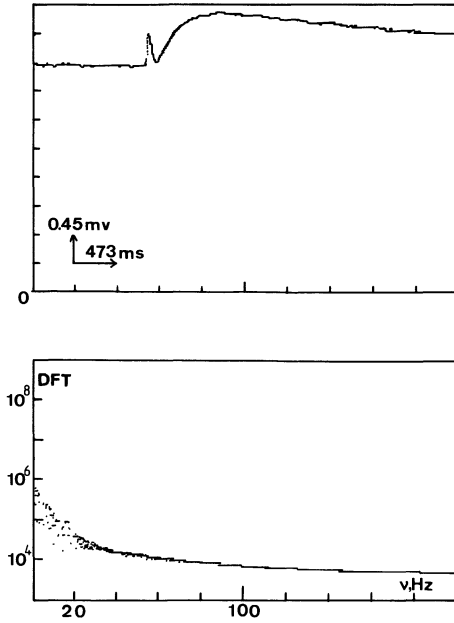


Fig. 10. — Transient signal from figure 8a filtered of by the use of the recursive filter having parameters as in figure 9 and its DFT computed for the sampling period $T_s = 2.5$ ms.

However, in certain limits, the signal frequency spectrum can be shifted into low frequency by the selection of a greater thickness of the sample.

4. Diffusivity determination.

4.1 THE FINITE PULSE EFFECT. — When the duration of the pulse τ is comparable to the characteristic rise time t_c of the rear face temperature

$$t_c = \frac{L^2}{\pi^2 a} \quad (11)$$

the increase of that temperature will be delayed (in comparison with the increase due to the instantaneous pulse) and the coefficient equal 1.38 in equation (7) will be greater. Such a situation takes place when the sample are thin and/or when their thermal diffusivity has a high value. The coefficient 1.38 in equation (7) is accurate within about 1 % for $t_c > 50 \tau$ [17].

The effect of delaying depends on the duration of the pulse as well as on its shape. The models corresponding to different shapes of pulses have been developed by Taylor and Cape [7] for saw-tooth and square wave shape, by Taylor and Clark III [8], Heckman [18] for triangular shape and by Larson and Koyama [20] for exponential shape.

The models developed lead to corrections of the model valid for instantaneous pulse (Parker *et al.* [1]) and therefore to determination of the actual value to the coefficient equal to 1.38 in equation (7). The methods used for the corrections calculation includes the adjustment of the effective irradiation time using

the centre of gravity of the pulse [20] or the analytical derivation of the shape of the pulse together with the evaluation of integral of the product of this shape by a function corresponding to the response to a unit pulse (Green function) — as did Heckman [18] or Cape and Lehman [21]. The integral could be determined numerically as did Koski [6] using discretized laser pulse power vs. time shape. The numerical procedure is, in our opinion, recommended when the pulse shape is registered and analysed each time the diffusivity experiment is made. As we do not have this facility, we have decided to determine the average pulse shape which corresponds to the actual laser parameters (see chapter 2.1) and to determine the convolution integral analytically.

The typical shape of the pulse given by our Nd : glass is given in figure 2. It can be approximatively decomposed into two square waves, so the resulting pulse power evolution with time is given by the function $F(t)$ such as :

$$F(t) = \begin{cases} h & \text{for } 0 < t < \gamma \cdot \tau, \\ l & \text{for } \gamma \tau < t < \tau. \end{cases} \quad (12)$$

The ratio $\frac{h}{l}$, determined experimentally, and unity of heat input condition

$$\int_0^t F(t) dt = 1 \quad (13)$$

are sufficient to determine the rear face temperature $T_c(L, t)$ of the sample :

$$T_c(L, t) = \int_0^t F(t') T(L, t - t') dt', \quad (14)$$

where $T(L, t)$, given by equation (6), is the response function to a unit pulse. As this function corresponds to a model of thermal insulated sample the solution of the integral (14) is valid only for such conditions too.

The parameters of the pulse have been determined experimentally using the method described in § 2.1 moving the optical fibre by steps of 1 mm along the pulse diameter (equal to 8 mm). In each such position the pulse parameters correspond to the mean value obtained for 5 successive laser shots. Then, for every laser charge voltage, the mean values of the parameters, determined finally for 40 shots (8 positions \times 5 shots in each position), have been found. Table III gives typical pulse parameters.

The integral (14) has been derived analytically-taken the upper limit $t \geq \tau$ and the corrections in the diffusivity determination have been made the following way :

1° the set of pulse parameters $\left(t, \gamma, \frac{h}{l}, \frac{\tau}{t_c}\right)$ permit to find the value $\frac{t_{0.5}}{t_c}$

Table III. — Parameters* of the pulse for some laser charge voltages.

Laser voltage V	τ μs	δ_τ	γ	δ_γ	$\frac{h}{l}$	$\frac{\delta_h}{l}$
1 300	230	22.5	0.347	0.0652	3.04	0.394
1 600	344	9.57	0.156	0.0196	4.49	0.501
2 000	429	8.71	0.157	0.0147	3.64	0.342

* δ is the standard deviation taken for 8 different positions along the pulse diameter.

2° the transformations

$$\frac{\frac{\tau}{t_c}}{\frac{t_{0.5}}{t_c}} = \frac{\tau}{t_{0.5}} \quad \text{and} \quad \frac{t_{0.5}}{1.38 t_c} = \frac{a_c}{a}$$

are made to eliminate the, experimentally unknown, t_c value.

Finally, a corrected diffusivity value a_c can be found from the curves similar to that shown in figure 11 when one knows experimentally : the laser voltage (and therefore all the pulse parameters) and $t_{0.5}$. Figure 12 shows an illustration of the difference between the theoretical evolution of the rear face temperature for the case of instantaneous pulse ($\frac{\tau}{t_c} = 0.2$) when the pulse parameters correspond to a laser charge voltage of 1 300 V.

4.2 THE HEAT LOSSES CORRECTIONS. — The solution of the heat diffusion equation, when heat losses should be considered, has been given by Watt [22]. He has taken an instantaneous pulse assumption and supposed a one-dimensional heat flow together with the heat losses at front and rear faces⁽³⁾. The rear face temperature evolution $T_n(L, t)$ is given by the following equation :

$$T_h = T_{\max} \sum_{n=1}^{\infty} Y_n(0) Y_n(L) \exp\left(\frac{\kappa_n^2 t \cdot a}{L^2}\right), \quad (15)$$

where

$Y_n(x) =$

$$\frac{\sqrt{2(\kappa_n^2 + Z_2^2)} \left[\kappa_n \cos\left(\frac{\kappa_n x}{a}\right) + Z_1 \sin\left(\frac{\kappa_n x}{a}\right) \right]}{\sqrt{(\kappa_n^2 + Z_1^2)(\kappa_n^2 + Z_2^2 + Z_2) + Z_1(\kappa_n^2 + Z_2^2)}}$$

in which $Z_{1,2} = \frac{4 L \sigma \varepsilon T_{\text{av}}^3}{\lambda}$, σ being the Stephan-Boltzmann constant, ε the sample emissivity, λ the

⁽³⁾ In typical experimental conditions the lateral surface of the sample is much smaller than that of the rear and front faces thus it is reasonable to neglect the lateral losses.

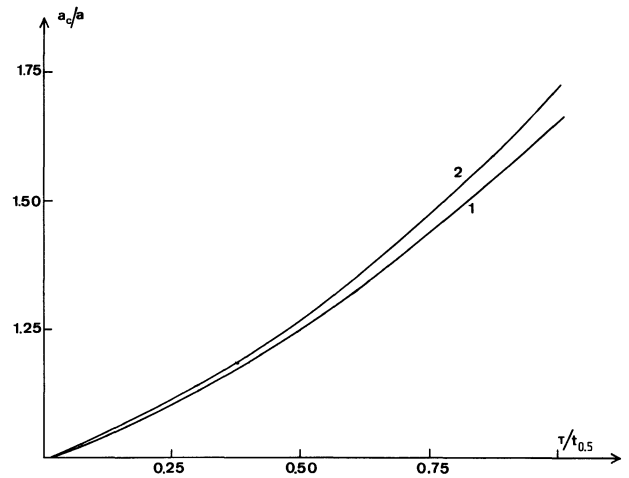


Fig. 11. — Evolution of the ratio a_c/a (a_c being the corrected diffusivity for the finite pulse duration) vs. ratio $\tau/t_{0.5}$ for a laser pulse parameters corresponding to the laser charged respectively under 1 300 V-1 and 2 000 V-2.

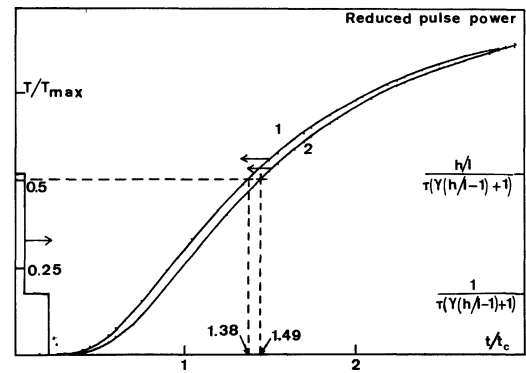


Fig. 12. — Theoretical evolution of the sample rear face temperature resulting from instantaneous pulse-1 and finite duration pulse $\tau/t_c = 0.2-2$. Pulse parameters correspond to the laser charged under 1 300 V.

thermal conductivity, T_{av} is the average temperature of the sample (by taking the mean value of the samples temperature evolution with time beginning at the moment when the pulse is striking the sample front face), indexes 1 and 2 correspond to sample front and

back face and $\kappa_n (n = 1, 2, 3, \dots)$ are the positive roots of the transcendental equation

$$(\kappa - Z_1 Z_2) \tan \kappa = \kappa(Z_1 + Z_2).$$

The solution (15) has been analysed by numerous authors [18, 21, 23, 24, 26, 27] and, as for the finite pulse effect, the influence ⁽⁴⁾ of heat loss parameter Z onto the coefficient, equal to 1.38 in equation (7), has been investigated. The methods used for the evaluation of this coefficient are generally based on the analysis of the theoretical curve given by equation (15) — either for the heating [18, 24, 26, 27] or for the cooling part [18, 21, 23]. The analysis of the heating part of the curve requires the precise knowledge of the evolution of experimental temperature vs. time from the beginning of the curve. However experimentally the precision of these points is low because of the « flash-by » phenomenon, i.e. signal distortion due to the influence of the reflected laser flash light onto the temperature evolution (see e.g. Fig. 10a). This distortion is especially important if the sample is thin and/or has high diffusivity and even the points $t_{0.3}$ and $t_{0.4}$ may be perturbed. That is why we have decided to use the cooling part of curve analysis.

To compare the different methods we have used, as test material, the sample of POCO graphite (type AXM – 5Q1) having a thickness $L = 0.0096$ cm and we have compared the results of the diffusivity, obtained using different corrections, to the standard values [25]. The comparison is shown in table IV. The results are in reasonable agreement with the standard data (within 10 %) and in good agreement (within 8 %), when one excludes the measurement at 1 090 K. The analysis of the results permits to conclude that it is recommended to apply the Heckman method which use several characteristic points of the temperature evolution, resulting in a better precision than the methods taking only one characteristic point (as the Cowan's one).

⁽⁴⁾ It is worth to underline that, when the finite pulse effects are considered, the coefficient is greater than 1.38 but with heat losses the coefficient value is less than 1.38.

5. The experimental set-up.

The previous analysis has been tested with the diffusivity apparatus shown in figure 13. The Nd : glass laser was used as a pulse source. The pulse was directed onto the sample *via* two copper mirrors, a beam splitter, a diaphragm and a silica window. The sample, being 8 mm in diameter, is hold, by the molybdenum fixation sketched in figure 14, into the tantalum furnace which permits to obtain temperatures up to 2 300 K. The furnace and sample's holder are closed into a vacuum bell-jar and the vacuum system permits to obtain a pressure of 10^{-6} torr. The thermal signal given by the rear face is focused by CaF_2 lens onto an InSb infrared detector (cooled by liquid nitrogen) in such a way that the detector sees the central part of the sample (about 4 mm of diameter). The small d.c. potential generated by the detector is reduced to zero using a voltage bias. The signal, transmitted through preamplifier and amplifier is introduced into the digital memory oscilloscope. The data from oscilloscope are sent to a small microcomputer, and stored before the analysis, described in the previous paragraphs, is made in order to determine the thermal diffusivity.

To test the apparatus and the described methods the diffusivity of the Armco iron has been determined (Fig. 15). The results are in reasonable agreement with the recommended data.

6. Conclusion.

The analysis of the problems due to the adaptation of the flash diffusivity method to a digitalized data acquisition system (DDAS) is carried out. The DDAS permits a systematic control of the pulse power evolution with time which is the base to consider the effects of finite pulse time and to determine the overall pulse during diffusivity measurement. The method of smoothing the experimental transient signal based on the digital Fourier transform is also presented. The experimental set-up for the diffusivity measurements together with the results of diffusivity of standard samples (POCO graphite and Armco iron) are the practical successful confirmation of the present analysis.

Table IV. — Diffusivity of the poco graphite (AXM-5Q1) corrected for the heat loss using the different methods.

Temperature K	a_{apparent} $\frac{\text{cm}^2}{\text{s}}$	a_{Heckman} $\frac{1/18}{\text{cm}^2/\text{s}}$	$a_{\text{Cape-Lehman}}$ $\frac{1/21}{\text{cm}^2/\text{s}}$	a_{Cowan} $\frac{1/23}{\text{cm}^2/\text{s}}$	a_{Agard} $\frac{1/25}{\text{cm}^2/\text{s}}$
1 090	0.184	0.148	0.179	-	0.162
1 216	0.172	0.152	0.162	0.164	0.162
1 392	0.147	0.131	0.134	0.139	0.132
1 426	0.143	0.130	0.133	-	0.129
1 670	0.128	0.118	0.121	0.118	0.112
1 739	0.117	0.101	0.103	0.103	0.108
1 960	0.117	0.0962	0.0956	-	0.0996

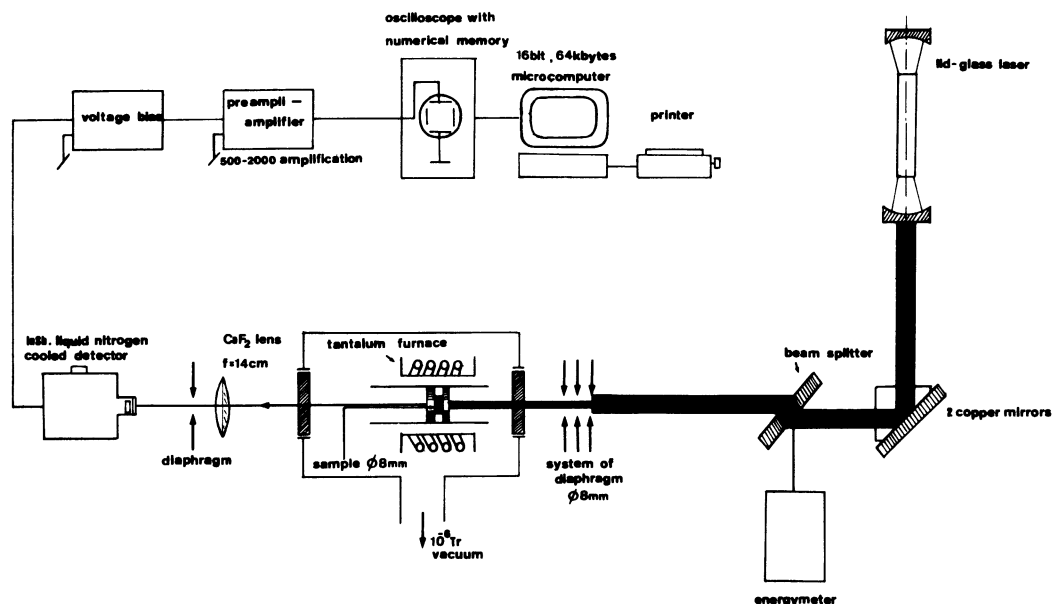


Fig. 13. — Sketch of the diffusivity measurements apparatus.

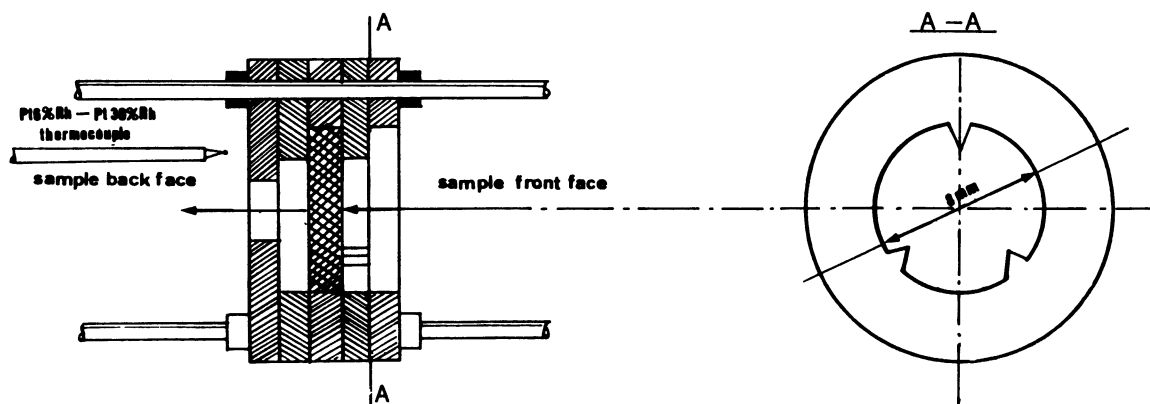


Fig. 14. — Details of the sample support.

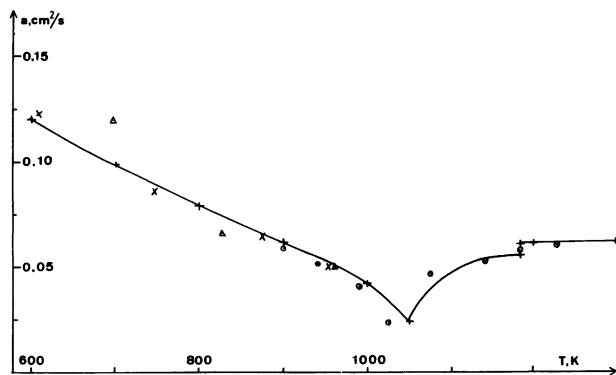


Fig. 15. — Diffusivity of the Armco iron, Δ-uncoated sample of thickness $L = 0.1$ cm, × -uncoated sample of thickness $L = 0.2$ cm, O-sample of thickness $L = 0.1$ cm coated by a thin graphite coating, full lines-standard data [12].

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