Journal of Thermal Analysis, Vol. 30 (1985) 33–42

IN SITU LASER THERMAL ANALYSIS OF BULK MATERIALS

P. Cielo

INDUSTRIAL MATERIALS RESEARCH INSTITUTE, NATIONAL RESEARCH COUNCIL OF CANADA, BOUCHERVILLE, QUÉBEC, J4B 6Y4 CANADA

The application of thermal analysis techniques directly to the materials being processed could open up new opportunities in the fields of non-destructive testing and process monitoring on the industrial floor. In this paper, a convergent-thermal-wave technique is proposed for the measurement of thermal diffusivity when access to the material is restricted to one side only. Such a technique permits measurement of the thermal properties of samples of large or unknown thickness, as well as coated or stratified materials. A finite-difference numerical model is used to evaluate the possibilities and the accuracy offered by such a technique, and a laser-based experimental apparatus for the non-contact generation and monitoring of annular thermal waves is described.

Thermal analysis methods constitute a powerful tool for the research and analytical scientist. Thermogravimetry, differential thermal analysis and differential scanning calorimetry are now essential components of the analytical laboratory, while thermal conductivity and diffusivity measurements are increasingly performed in the thermophysics laboratory for quality control and material assessment purposes. The possibility of using similar techniques on the industrial floor is an attractive opportunity. Typical applications are the non-destructive evaluation of various materials in the different fabrication stages as well as during the service life, and the continuous monitoring of materials production or treating processes.

For this purpose, fast and practical techniques for the remote inspection of thermal properties are required. Such techniques should require no sample preparation and should be applicable in hostile environments such as a hot-extrusion machine or a ceramic-sintering furnace. Laser-flash techniques [1, 2] with infrared temperature sensing are potentially adaptable to such applications, because they are non-contact and fast. However, such techniques require samples of given shape and thickness, which are flash-heated on one surface while the arrival of the thermal wave is monitored on the opposite surface.

One-side techniques using a laser beam to heat a small area on the surface of a bulk sample and monitoring the thermal propagation at a given distance *d* from the heated area on the same surface (see Fig. 1a) have recently been developed [3–5]. One problem involved in such techniques is the limited amplitude of the detected temperature signal far from the heated spot [6]. On the other hand, decreasing the distance *d* also decreases the accuracy, because of such hardly predictable parameters

John Wiley & Sons, Limited, Chichester
Akadémiai Kiadó, Budapest

as the intensity distribution of the laser beam within the irradiated area, the lack of uniformity of the infrared sensor sensitivity across its sensitive area, and the variations of the surface emissivity and absorptivity within the irradiated and the probed areas.

This paper presents a converging-thermal-wave technique which provides an increased signal amplitude without reducing the distance *d* and without overheating the irradiated area. The basic idea is demonstrated in Fig. 1. Figure 1a shows the conventional approach, and Fig. 1b the converging-wave approach, where an annular area is heated and the temperature is probed in the centre of the annulus, where the converging thermal wave reaches a conveniently high amplitude. The possibilities of such a method are demonstrated in the next section through a finite-difference axisymmetric model. An optical technique providing focused annular irradiation without loss of intensity is described in the following section.

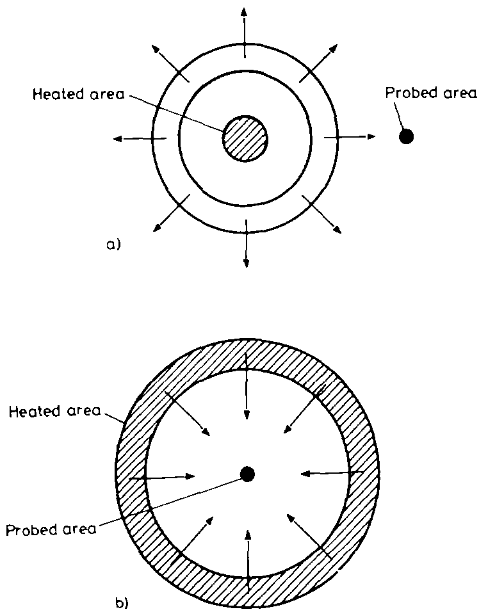

Fig. 1 Basic idea of the convergent-wave technique for the thermal inspection of bulk materials with access from one side only; (a) probing a diverging thermal wave; (b) probing a converging thermal wave

Numerical analysis

A finite-difference, axisymmetric numerical model was used to examine the propagation of the thermal wave in the irradiated material in the case of either a diverging or a converging configuration and under conditions of either pulsed or sinusoidally modulated laser irradiation. The model uses the explicit iterative approach [7] to obtain a time-resolved solution of the heat diffusion equation:

$$\frac{\partial T}{\partial t} = \frac{1}{\rho c} \operatorname{div} (K \operatorname{grad} T) \tag{1}$$

where T is the temperature, t the time, ρ the density and c the specific heat of the heated material.

Typically, 400 toroidal elements are used to model a 2 cm radius, 2 cm deep volume. The model accepts temperature-dependent thermal conductivity and specific heat data, as well as phase changes which are accounted for through the use of an internal energy matrix.

The boundary conditions are defined as follows. Radiation and convection losses are assumed for the surface elements, while the condition of continuity $T \propto 1/r$ is assumed at the lateral and bottom surfaces. Such a choice is justified by the observation that the temperature field far from the heat source tends to the field of a continuous point source [5]:

$$T(r) = \frac{P}{2\pi K r} \tag{2}$$

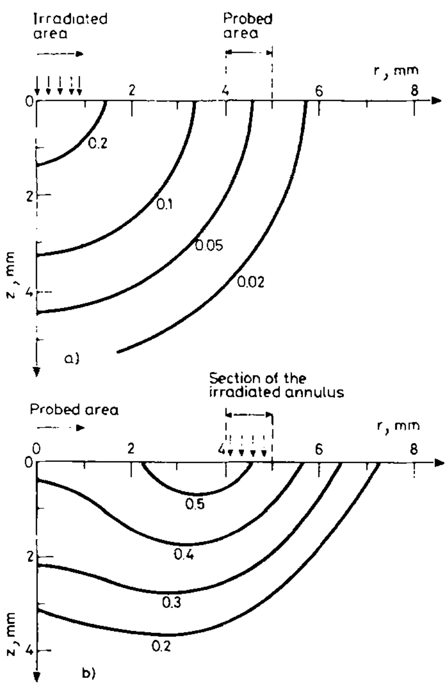

Fig. 2 Cross-section of the computed thermal distribution within the irradiated material 0.2 sec after the beginning of the 0.03 sec heat pulse; (a) diverging-wave configuration; (b) converging-wave configuration. The isotherms are in °C

where r is the radial distance from the centre of the heated area and P is the injected heat flux.

The temperature distribution within a uniform sample heated by a 0.03 sec annular laser pulse is shown in Fig. 2. The thermal properties are those of carbon steel. It can be seen that the diverging wave (Fig. 2a) spreads three-dimensionally, resulting in a particularly low temperature level when the thermal front reaches the position of the probed surface. On the other hand, the converging-wave configuration (Fig. 2b) produces a higher temperature level on the probed area at the centre of convergence. In both cases, an equal pulse energy density of 3 J/cm^2 was assumed, resulting in a much larger total energy for the annular irradiation as compared to the spot irradiation configuration. Such a choice provides the same maximum temperature of the irradiated surface, which is usually the limiting factor to the power of the heating laser.

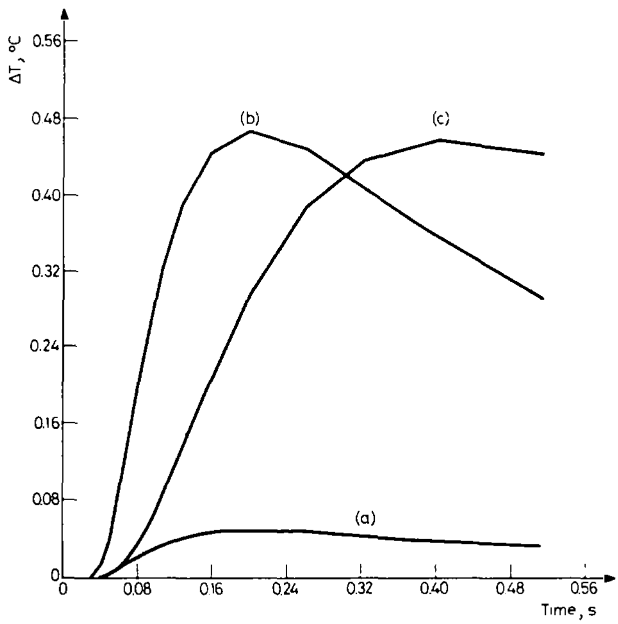

Fig. 3 Temperature histories of the probed area, in the diverging-wave configuration (curve (a)) and in the converging-wave configuration (curve (b)). Curve (c) is obtained with the same configuration as curve (b), but with half the thermal diffusivity

Figure 3 shows some temperature history curves of the probed area, in the case of (a) the diverging-wave configuration (temperature history of the probed area in Fig. 2a) and (b) the converging-wave configuration (probed area in Fig. 2b). The thermal parameters and heat source are the same as for Fig. 2. The signal is much higher in the case of the converging-wave. Curve (c) corresponds to the same configuration as curve (b), but assuming a thermal diffusivity half that for curve (b).

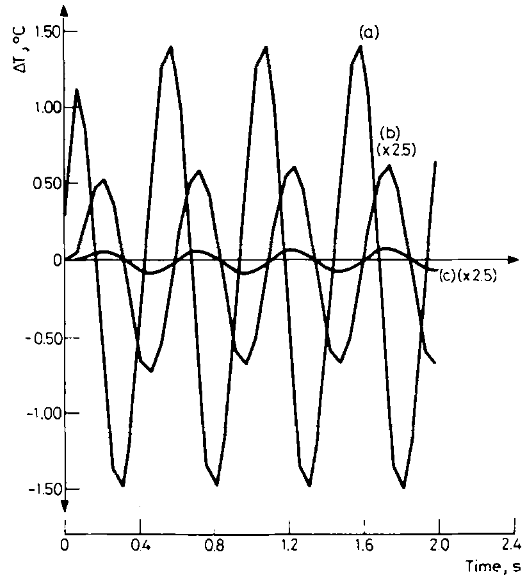

Fig. 4 Thermal histories in the case of a sinusoidally modulated laser beam; (a) converging-wave temperature of the irradiated area; (b) converging-wave temperature of the probed area; (c) temperature of the probed area in the diverging-wave configuration

Knowing the radius of the annulus, an unknown diffusivity can be evaluated from the position of the peak temperature or of the half-risetime of such a curve.

An alternate approach for the measurement of the diffusivity is shown in Fig. 4. In this case, a sinusoidally modulated annular source is assumed, providing a temperature fluctuation of the heated annular surface which is given by curve (a). The induced thermal fluctuation in the centre of the annulus is given by curve (b). Only the AC portion of the signal is shown in such curves. The thermal diffusivity can be obtained from a measurement of the phase delay of the detected signal, a technique similar to the diverging-thermal-wave approach [1, 3, 6]. Again, the conventional diverging-wave configuration would provide a signal level (curve (c)), much smaller than the converging-wave configuration (curve (b)).

Experimental apparatus and discussion

In order to produce an annular-shaped heated area, one could use an annular mask, but this would introduce a significant energy loss. High-speed rotating mirrors are cumbersome and limited in service life.

Figure 5 shows the approach which is being investigated at our Institute. An axicon (lens of conical section) refracts the laser beam into an annular beam impinging on the surface. A photo of the focused annular beam is shown in the insert. An infrared de-

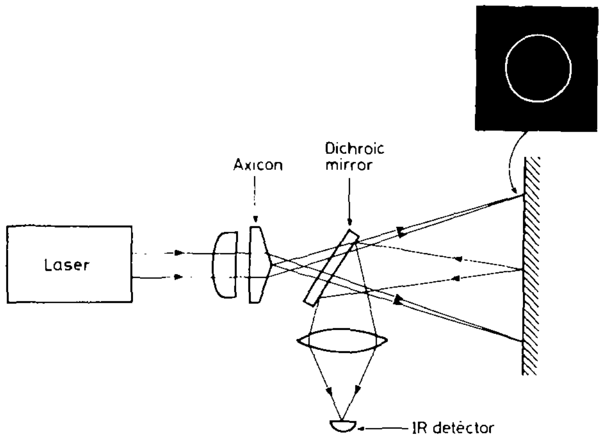

Fig. 5 Scheme of the optical apparatus for producing an annular-shaped heated area (shown in the insert). A combination of a lens and an axicon produces an annular laser beam which is focused on the surface. The resultant thermal fluctuation in the centre of the annulus is detected by a focused infrared detector

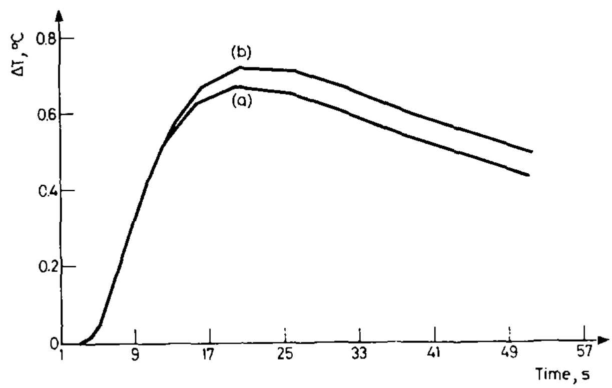

Fig. 6 Effect of the surface losses on a low-diffusivity sample heated by a 5 mm radius annular source; (a) thermal history curve computed with surface losses; (b) same curve assuming no surface losses

tector monitors the temperature variation in the centre of the annulus through a dichroic mirror, which transmits the laser wavelength but reflects the infrared radiation. Tests on different materials are under way at our Institute using such an apparatus.

The accuracy of the converging-wave technique can be evaluated using the numerical model. Figure 6 shows the effect of the emissivity- and ventilation-dependent radiation and convection losses on the detected signal. A low thermal diffusivity ($\alpha = 0.0015$ cm^2/s) material was assumed to enhance the effect of surface losses.

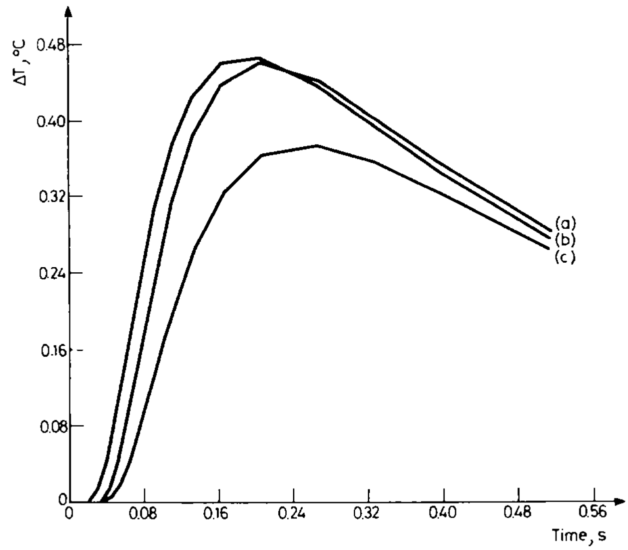

Fig. 7 Effect of positioning and focusing errors on the detected signal; (a) ideal conditions; (b) focusing error for the detector; (c) positioning error for the heating beam (see text)

Curve (a) was obtained under standard surface loss conditions, while curve (b) was obtained assuming no surface loss. Although the surface losses have a definite effect in this case, neither the position of the peak temperature nor the half-risetime appear to be significantly modified by this factor. Even less significant effects are obtained with higher conductivity materials.

Figure 7 shows the effects of positioning and focusing errors of the annular laser beam and of the IR detector. Curve (a) corresponds to a well-positioned configuration: a 1 mm wide, 5 mm radius annulus with the IR detector focused on a 1 mm radius central spot. Curve (b) was obtained in the case of an unfocused IR detector giving a 1.5 mm radius probed spot, while curve (c) corresponds to a laser positioning error producing a 2 mm wide, 6 mm radius annulus. Both such errors produce a significant shift in the position of the peak temperature and the half-risetime. Indeed, the approximate relation $t \sim d^2/4\alpha$ between the thermal propagation time t, the distance d between the heated and the probed area, and the thermal diffusivity α, leads to an error of 2% in the evaluated diffusivity for every 1% variation of the distance d.

Although the positioning errors assumed in Fig. 7 should be avoidable by a normally careful operator, an equivalent error would be obtained if the emissivity of the material varies within the probed and the heated areas. In order to avoid such errors, one can: (1) increase the distance d, at the expense of a lower signal amplitude and longer time response; or (2) reduce the width of the annulus and of the probed spot, within the limits imposed by the aperture of the optics, by the maximum temperature allowed for the heated surface, and by the sensor detectivity, which is proportional to the diameter of the sensitive area. Typical inaccuracies of the order of 5 to 10% in the diffusivity measurement are thus expected from this source of error.

Finally, a source of error related to the optical penetration depth is analyzed in Fig. 8. Curve (a) was obtained by assuming a surface absorption of the heating laser

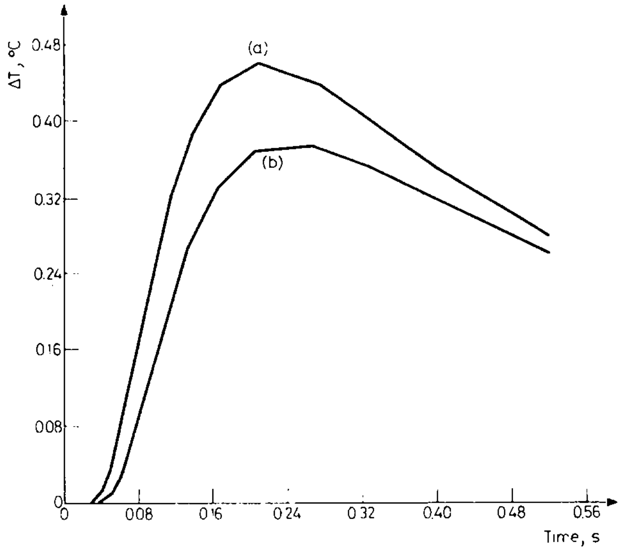

Fig. 8 Error produced by an unknown optical penetration in the material; (a) material perfectly opaque; (b) penetration depth of 3 mm for the heating beam

and a surface emission of the thermal radiation, while curve (b) corresponds to a surface emission but a laser penetration depth of 3 mm. Such a parameter can be readily measured, however, by inspecting the surface being heated by a short pulse [8]. As Fig. 9 shows, the slope of the curve in a log-log plot at the end of the pulse is strongly related to the optical penetration depth.

Conclusion

A converging-wave technique is described for the analysis of materials accessible from one side only. Such a technique provides an evaluation of the thermal diffusivity of the material with limited signal loss. The value of either the specific heat or the thermal conductivity of the material can be inferred from such a measurement. An optical apparatus is described for the non-contact thermal inspection of different materials by this technique.

* * *

The technical assistance of M. Lamontagne of IMRI is gratefully acknowledged.

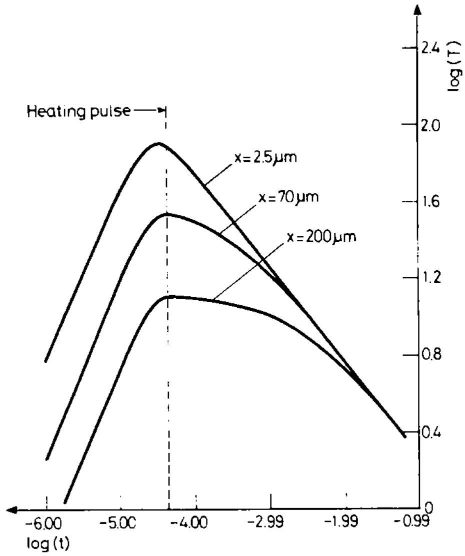

Fig. 9 Method for determining the optical penetration depth in the material. The three curves correspond to the detected surface temperature after a short laser pulse, for different penetration depths x. The penetration depth can be evaluated from the slope of the curve at the end of the laser pulse

References

1 Y. S. Touloukian et al., Thermophysical Properties of Matter, vol. 10, IFI/Plenum, 1973.
2 A. Degiovanni, Rev. Gen. Therm. Fr., 185 (1977) 420.
3 J. Jaarinen and M. Luukkala, Proc. 3rd Top. Meeting on Photoaconst. and Phototherm. Spectr., Paris, Apr. 5–8, 1983.
4 A. Rosencwaig, J. Opsal and D. L. Willenborg, Appl. Phys. Lett. 43 (1983) 166.
5 P. Cielo, Proc. 4th Symp. on NDE, San Antonio, Texas, Apr. 19–21, 1983.
6 H. S. Carslaw and J. C. Jaeger, Conduction of Heat of Solids, sect. 4.4, Oxford Un. Press, Oxford, 1959.
7 L. C. Thomas, Fundamentals of Heat Transfer, chapt. 3, Prentice-Hall, Englewood Cliffs, 1980.
8 A. C. Tam and B. Sullivan, Appl. Phys. Lett., 43 (1983) 333.

Zusammenfassung — Die direkte Anwendung thermischer Analysenverfahren auf darzustellende Materialien könnte neue Möglichkeiten auf dem Gebiet der nichtzerstörenden Materialprüfung und der industriellen Prozeßüberwachung bieten. In diesem Artikel wird eine Konvergenzthermowellen-Technik zur Messung des thermischen Diffusionsvermögens vorgeschlagen, die auch dann anwendbar ist, wenn das Material nur von einer Seite her zugänglich ist. Diese Technik ermöglicht die Messung thermischer Eigenschaften von Porben unbekannter Größe oder Dicke sowie die von belegten oder beschichteten Materialien. Ein numerisches endliches Differenz-Modell wird zur Bewertung der durch solch eine Technik gebotenen Möglichkeiten und deren Genauigkeit herangezogen. Eine auf Laser basierende Versuchsapparatur zur Nicht-Kontakt-Erzeugung und Aufzeichnung von ringförmigen thermischen Wellen wird beschrieben.

Резюме — Применение методов термического анализа непосредственно к анализируемым материалам открывает новые возможности в области недеструктивного анализа и процесса контроля в промышленном масштабе. Метод концентрической термической волны предложен для измерения коэффициента термической диффузии, когда доступ к материалу органичен только одной стороной. Метод позволяет проводить измерения термических свойств образцов большой или неизвестной толщины, а также слоистых материалов или материалов с покрытиями. Для определения возможностей и точности метода предложена модель численной конечной разности. Описана на основе лазера экспериментальная установка для неконтактного генерирования и контроля кольцевых термических волн.