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AN INTERFEROMETRIC METHOD FOR MEASUREMENT OF THE THERMAL CONDUCTIVITIES OF SOLIDS

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An interferometric method is described for the determination of small deformations of a solid surface when heated by a c.w. gaussian laser beam, from which temperature increase and thermal parameters can be derived. A discussion of the method is presented and applied to the measurement of thermal conductivity, for two different situations of an unrestrained or restrained sample heated with a c.w. laser. Experimental results are finally given for a semiconductor crystal of Si.

Thermal conductivities of solid samples can be measured through determination of the dilatation of the sample when heated with a laser. Several geometries can be used for this purpose; here we consider the case of heating with a continuous laser. Dilatation can be measured with a simple interferometric method, which allows the determination of small displacements of the order of less than one thousand Ångstroms. In the case of heating with a c.w. laser, after a short time, if the sample is not too large, the temperature is nearly uniform throughout it, and dilatation is simply proportional to the dilatation coefficient and to the temperature increase. As the temperature increase is a function of the thermal conductivity, the method allows measurement of this latter quantity if the other material parameters are known.

Below we describe first the interferometer used and two possible heating geometries: one with an unrestrained slab, and the other with a slab with its ends axially restrained. Further, the principal mathematical relation is derived and example of application is discussed for a silicon sample. This example has been chosen because the thermal conductivity is well known for this material, and a comparison can be made between the known values of this quantity and the values derived from our measurements as a function of temperature.

Experimental set-up

The interferometer used is shown in Fig. 1. The beam from a He–Ne laser is made parallel by a telescope *T* and sent on to the main part of the interferometer which is a cube S, built up from two roof prisms faced with each other, with the contact surface Su partially reflecting. The laser beam is therefore split into two beams 1 and 2, which are reversed by a corner prism P and by reflection at the specular surface of the sample Se through a lens L, respectively. They are then superposed on the detector D.

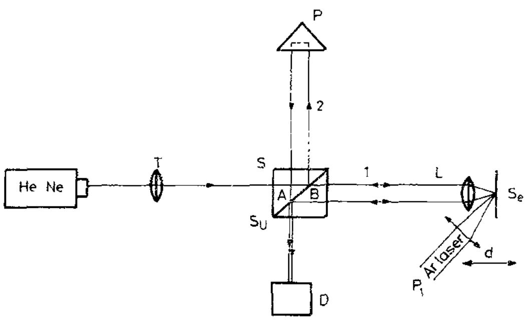

Fig. 1 Interferometer

The sample is heated by a powerful laser P_1. Two different mountings of the sample can be used; without rigid surface restraining, or with rigid surface restraining. In both cases, if the light intensity in the two split beams is the same, I, the intensity at the detector, I_D, can be written as

$$I_D = 2I(1 + \cos \Delta\Phi) \tag{1}$$

where $\Delta\Phi$ is the phase difference between the beams:

$$\Delta\Phi = \frac{2\pi}{\lambda}(d_1 - d_2) + \delta\Phi \tag{2}$$

d_1 and d_2 being the distances travelled from point A to point B through the two paths, respectively, and taking into account the phase shift at the reflections. The heating laser beam (Ar^+ laser) impinges on the surface Se in the same point which is inspected via the interferometer. The dilatation $S(t)$ of the surface produces a phase shift

$$\delta(\Delta\Phi) = \frac{2\pi}{\lambda} 2S(t) \tag{3}$$

The number of fringes detected by D and recorded on an X–Y plotter gives measurement of $S(t)$. If a fifth of the fringes can be appreciated at $\lambda = 6328$ Å, a minimum change of $\Delta l \cong 320$ Å is measured. If a great number of fringes pass through the detector, there is no difficulty in counting them, provided they move slowly enough.

Therefore, large displacement of the order of several microns can easily be measured with great precision.

With this disposition the vertical displacement of the centre of the heated spot is measured in a large range of values, and its value can be connected with the thermal dilatation of the material and the temperature distribution in the sample.

Theoretical relation between dilatation, time and temperature

The problem of heating and subsequent displacement of a solid slab of thickness l_0, illuminated on one side by a gaussian c.w. laser beam, is fully treated elsewhere [1].

The laser intensity is given by

$$I(r) = I_0 \exp\left(-\frac{2r^2}{W_0^2}\right) \tag{4}$$

where W_0 is the laser spot.

In the following we use the thermoelastic quasistatic formulation [2]. First, the temperature distribution at the centre of the laser spot is derived, starting from the Fourier equation for heat conduction in solids [3], and using the 'idea' of an instantaneous point source of heat liberated at time $t = 0$.

In the case of a slab with thickness $l_0 < \sqrt{\chi t}$ (χ is the thermal diffusivity), the expression for the temperature at the centre of the laser spot is very simple, and can be derived in the cases of slab in air or in vacuum (for a slab with transversal dimensions much larger than l_0).

In vacuum it is

$$\underset{\text{cont. gauss.}}{T(0,t,0)} = \frac{I_0(1-R)W_0^2}{4K(T)\,l_0} \ln\left(\frac{4\chi t + W_0^2}{W_0^2}\right) \tag{5}$$

where $K(T)$ is the thermal conductivity, χ is the thermal diffusivity, W_0 is the laser spot, and R is the reflectivity.

The temperature in air can be written as

$$\underset{\text{cont. gauss.}}{T(0,t,\bar{k}^{-2})} = \frac{I_0(1-R)W_0^2}{4K(T)\,l_0} \exp\left(\frac{\bar{k}^{-2}W_0^2}{4}\right)$$

$$E_1\left(\frac{\bar{k}^{-2}W_0^2}{4}\right) - E_1\left[\left(\frac{\bar{k}^{-2}W_0^2}{4}\right)\left(\frac{4\chi t}{W_0^2}+1\right)\right] \tag{6}$$

where E_1 is the exponential integral function, and $\bar{k}^{-2} = 2H/Kl_0$ represents the air losses, H being the air conductance.

From Eqs (5) and (6) it is apparent that after a very short time the temperature distribution is constant through the thickness of the slab.

When the slab is unrestrained (for example the slab is mounted with elastic and thermally isolated tongs), the thermoelastic theory yields a very simple expression

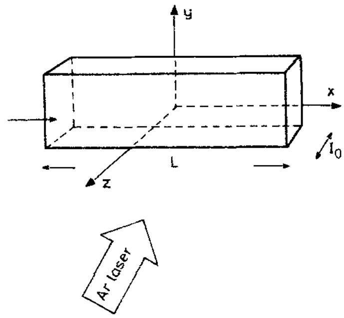

Fig. 2 Geometry for the restrained slab

for the vertical displacement at the centre of the laser spot ($r = 0$), which can be written as

$$S(t) \cong \alpha(1+\nu)\Delta T l_0 \tag{7}$$

where α is the thermal dilatation coefficient, ν is the Poisson coefficient, and ΔT is the temperature increase.

When the sample is heated in air, air heating must be taken into account, which produces a perceptible fringe displacement [1].

When the sample has both ends rigidly fixed in the axial direction (see Fig. 2), its vertical displacement can be derived in the approximation of 'small deflection' theory as

$$S_T(t) = S(t) + D(t) \tag{8}$$

where $S(t)$ is given by Eq (7) and [4]

$$D(t) \cong \frac{2L}{\pi}\sqrt{\frac{|P_T - P_{cr}|}{AE}} \tag{9}$$

where $D(t) = 0$ for $P_T < P_{cr}$, L is the transversal length of the slab, A is the cross-section, E is Young's modulus, $P_{cr} = \pi^2 EAl_0^2/3L^2$ (critical load), and $P_T \sim AE\alpha\Delta T$ in the approximation of uniform heating of the slab (this approximation is correct if the transversal dimension also fulfils $L < \sqrt{\chi t}$). From Eq. (9) we have

$$D(t) \sim \frac{2L}{\pi}\sqrt{\alpha\Delta T - \frac{\pi^2 l_0^2}{3L^2}} \tag{10}$$

for $P_T > P_{cr}$), and the total displacement, 'seen' by the interferometer is

$$S_T(t) = \alpha(1+\nu)\Delta T l_0 + \frac{2L}{\pi}\sqrt{\alpha\Delta T - \frac{\pi^2 l_0^2}{3L^2}} \tag{11}$$

Results and discussion

We have applied the preceding discussion to the simple case of a Si sample, by using a c.w. Ar^+ laser as a heating source. Figure 3 illustrates a typical run, showing the fringe movement from the time at which the Ar^+ laser was switched on, in the case of an unrestrained sample in vacuum, for a laser power $P = 3$ W.

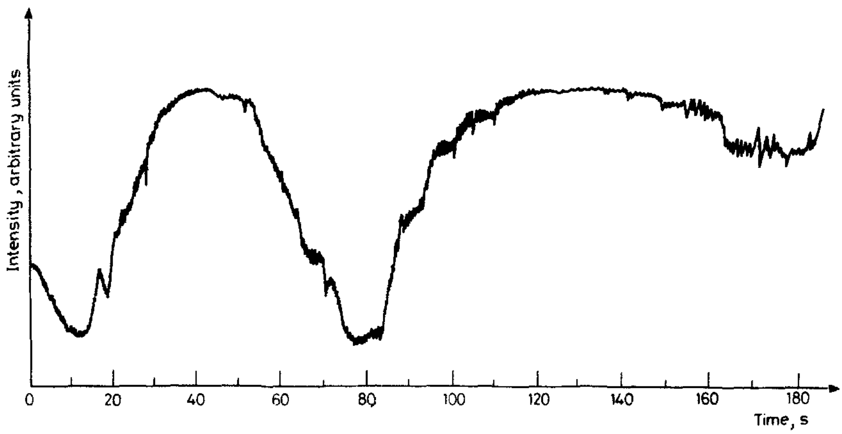

Fig. 3 Fringe movement with sample unrestrained in vacuum ($P = 3$ W)

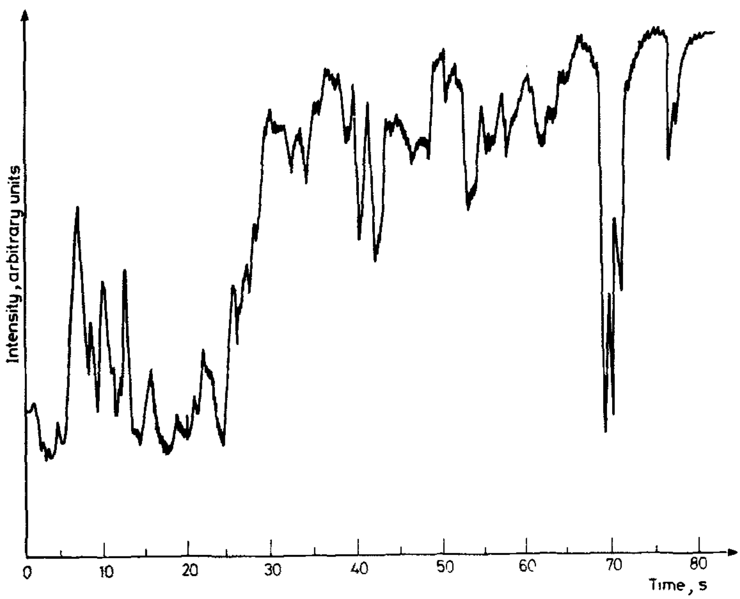

Fig. 4 Fringe movement with sample unrestrained in air ($P = 1.8$ W)

The effect of air heating is evident in Fig. 4, obtained with the sample unrestrained in air. In this case, a few seconds after the heating is began, the surface is already warm enough to heat air in its vicinity. The highly non-uniform thermal gradient produces a turbulent movement of the air, with consequent large fluctuations of the refractive index. These fluctuations appear in the interferometer as large fluctuations in the positions of the fringes, as depicted.

From runs such as those presented in Figs 3 and 4, the dilatation $S(t)$ as a function of time for an unrestrained slab can be calculated. Figure 5 shows this for different values of the Ar^+ laser power in vacuum; a comparison with theoretical results is also shown in Fig. 5. With a power $P = 3$ W, the estimated temperature increase is about 300° (see Fig. 6).

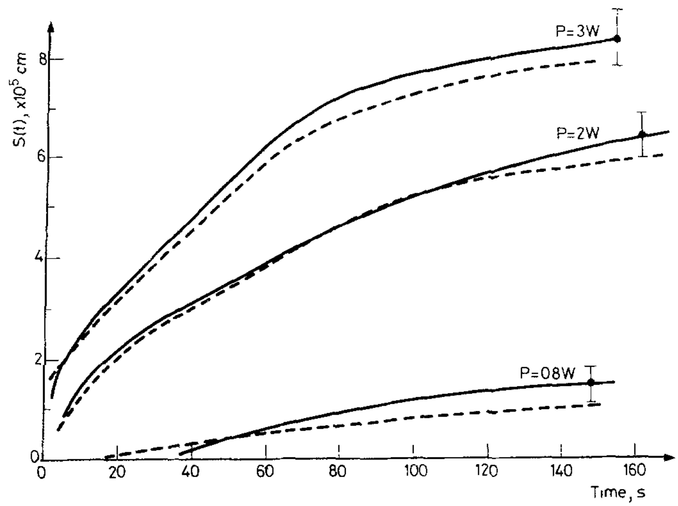

Fig. 5 Thermal dilatation for Si in vacuum; continuous curves are experimental data, broken curves are from theory

Figures 7a and 7b show fringe movements in the case in which the slab (Si with $l_0 \cong 50$ μm) has fixed ends, for powers $P = 0.1$ W and $P = 0.2$ W, respectively, in air. In this situation the displacement is due to the buckling of the heated slab. The estimated temperature increase is $\Delta T \sim 6°$ for $P = 0.1$ W in air, and $\Delta T \sim 9°$ for $P = 0.2$ W in air.

Figures 8a and 8b show the total displacement as derived from Figs 7a and 7b, respectively. The broken curves in Fig. 5 have been calculated by assuming a thermal conductivity behaviour with temperature

$$K(T) = \frac{A}{T - B} \tag{12}$$

with $A = 299$ W/cm and $B = 99$ K, as given in the literature [5]. The continuous curves in Fig. 8 have been calculated for a fixed value of $K = 1.5$ W/cm deg. Conversely, the curves in Figs 5 and 8 could be inverted to obtain ΔT and then $k(T)$. It can be shown, for example, that a change of 10% in the coefficient B in Eq. (12) would give the best fitting of the broken curves in Fig. 5 with the experimental points, as shown in Fig. 9.

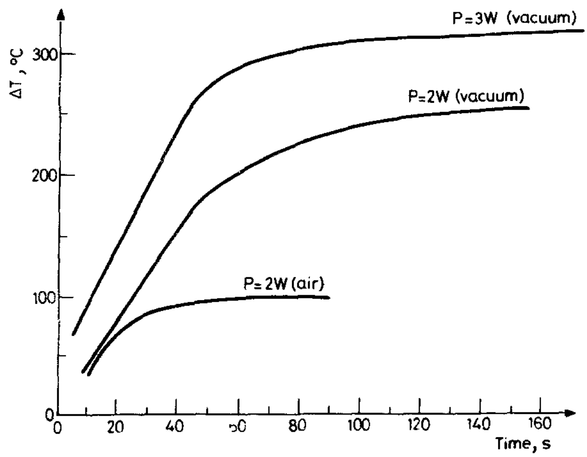

Fig. 6 Temperature values for different power values (unrestrained slab)

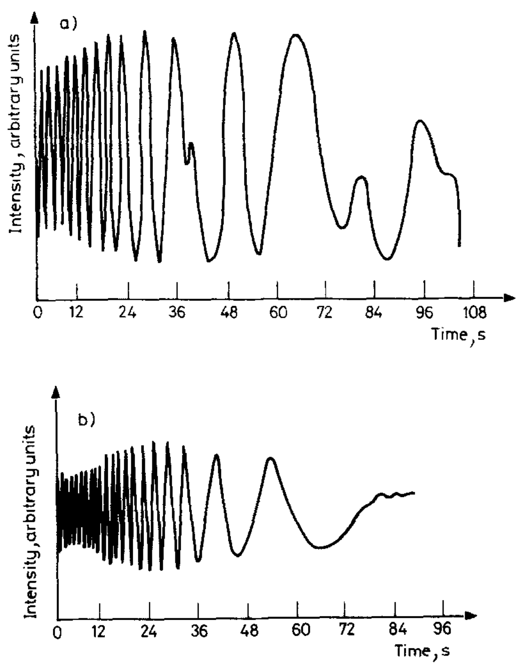

Fig. 7 Fringe movement for a restrained slab of Si ($l_0 = 50$ μm) in air. 7a) $P = 0.1$ W, 7b) $P = 0.2$ W

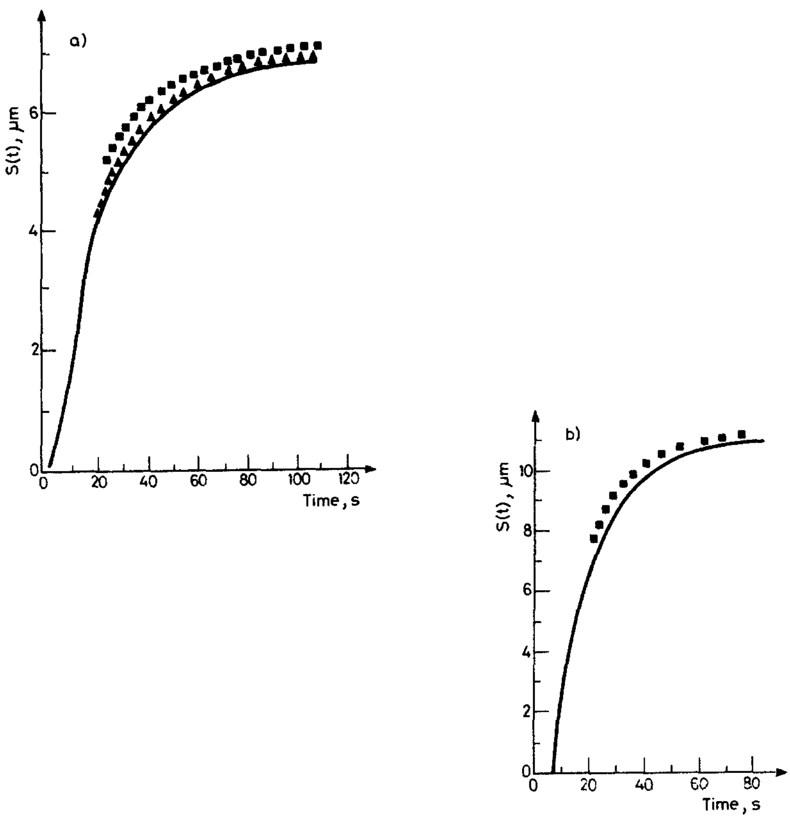

Fig. 8 Displacement of the restrained slab in air. 8a) $P = 0.1$ W, (■ ■ ■ curve is for $H = 10^{-3}$ W/cm^2 °C), (▲ ▲ ▲ curve is for $H = 5 \cdot 10^{-3}$ W/cm^2 °C), 8b) $P = 0.2$ W (■ ■ ■ curve is for $H = 5 \cdot 10^{-4}$ W/cm^2 °C)

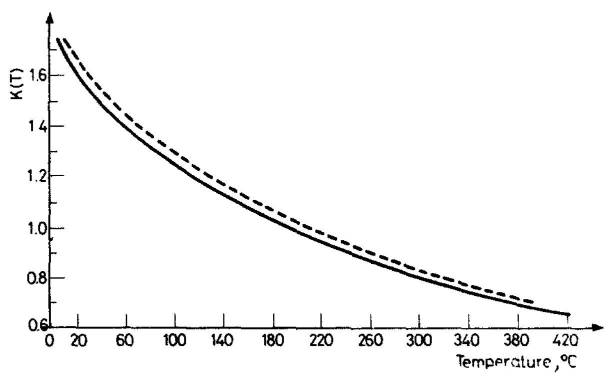

Fig. 9 Thermal conductivity as a function of temperature. Continuous curve as from literature; broken curve from theory

Conclusions

The above discussion and the examples show that it is possible to measure thermal conductivity as a function of the temperature of a solid sample by measuring the vertical displacement of the surface when heated with a laser.

As applied to silicon, for which thermal conductivity behaviour is well known, the method shows that the values derived by making the best fit of the theoretical curve with the experimental data give the expected thermal conductivity parameter within 10% of the values repoted in the current literature.

In vacuum there is no adjustable parameter, while in air a reasonable value for air losses has to be assumed.

Two different geometries have been discussed, which allow measurements to be made at nearly constant temperature (restrained sample) or as a function of the temperature (unrestrained sample).

Finally, it may be noted that the method can be used with energy-abosrbing specular surfaces.

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Zusammenfassung — Eine interferometrische Methode zur Bestimmung kleiner, beim Erhitzen mit einem Laserstrahl auftretenden Deformationen einer festen Oberfläche, aus denen die Temperaturerhöhung und thermische Parameter abgeleitet werden können, wird beschrieben. Die Methode wird diskutiert und ihre Eignung zur Bestimmung der Wärmeleitfähigkeit bei zwei verschiedenen Situationen einer mit einem c.w. Laser erhitzten "unrestrained" oder "restrained" Probe aufgezeigt. Experimentelle Ergebnisse werden für einen Silicium-Halbleiterkristall mitgeteilt.

Резюме — Описан интерферометрический метод определения небольщих деформаций твердых поверхностей при нагревании их лазерным лучём на основе которого может быть установлено увеличение температуры и термические параметры. Представлено обсуждение метода и применение его для измерения теплопроводимости образцов как подвергнутых нагреву лазерным лучём, так и не подвергнутых лазерному нагреву. Приведены экспериментальные результаты для полупроводникового кристалла кремния.