The measurement of the thermal conductivity of poly(methylmethacrylate) by Forbes' method, using infra-red temperature measurement

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The thermal conductivity of poly(methylmethacrylate) has been measured at and above room temperature by the well-known method of Forbes using modern infra-red imaging equipment to measure the temperature gradients. Forbes' method has hitherto been applied only to materials of high thermal conductivity and the method described extends its applicability to polymers and other poor thermal conductors.

Keywords Thermal conductivity; poly(methylmethacrylate); Forbes' method; infra-red; outer conductivity; insulators

Introduction

A knowledge of the thermal conductivity of plastics is of value in a number of contexts, as is that of the constant sometimes known, misleadingly, as the 'radiation constant', but better described as the 'outer conductivity' (i.e. the total heat lost in unit time from unit surface area per unit of temperature above background). For instance such values are important both in the theory[1] and experiment[2] in connection with crack propagation and also in the analysis of temperature change on subjecting a plastic material to tension, in particular with regard to the phenomenon of necking[3].

Many methods for the determination of thermal conductivity have been devised, mostly in the latter half of the nineteenth century. The simplest and perhaps the most useful are those based on conduction down a semi-infinite bar heated at one end, in that the variation of thermal conductivity with temperature may be obtained from a single such experiment. In particular the methods of Forbes[4,5] and that of Ångström[5,6] have been used to measure the thermal conductivity of metals but have not hitherto been used to measure thermal conductivity of poor conductors due to the experimental difficulties involved. The positioning of a sufficient number of conventional temperature sensing devices such as thermometers or thermocouples presents difficulties in view of the steep temperature gradients encountered and the effect the presence of closely-spaced sensing elements would have on the system. In the case of plastics there is an additional complication in that their softening points are comparatively low so that the maximum temperature to which the materials can be heated during an experiment is limited, in general, to less than 100°C. The advent of modern thermal-imaging techniques, however, presents the possibility of measuring the steep thermal gradients to be expected with materials of low thermal conductivity and low upper temperature limits without physical contact between the sensing device and the material under investigation. This work was designed to assess whether the basically simple method of Forbes could be applied to conventional plastics and other poor conductors by the use of thermal imaging techniques.

Experimental

The apparatus used was comparatively simple. A small heater consisting of an electrical heating element wound on a brass bobbin was used as the heat source and the temperature of the heater controlled by a variac. A quarter inch hole was drilled into one end of the bobbin into which the end of a rod of plastic could be inserted, a tight fit being ensured by machining. The temperature of one end of the rod could thus be adjusted. The further end of the rod was supported in a similar holder which was left at room temperature and the rod was sufficiently long to ensure that a considerable length of the rod remote from the heated end was maintained at room temperature. In these experiments a length of about twenty centimetres was found more than adequate to ensure this condition in which the rod can be regarded as semi-infinite.

In the initial form of the apparatus the heater and its cold counterpart were housed in a heavy brass cylinder with a view to protecting the test rod from draughts and to provide a background of uniform temperature into which the rod would radiate. A viewing slit 10 mm in width and 200 mm length was cut in this sleeve to provide a viewing port for the thermal imaging apparatus. It was quickly found, however, that the presence of this brass sleeve interfered with the operation of the thermal imaging apparatus and the brass tube was removed and replaced by a screen of stiff, matt-black paper forming a screen round approximately two thirds of the test-rod, the remaining opening serving as a viewing aperture for the detector. A drawing of this final form of the apparatus is shown in *Figure 1*.

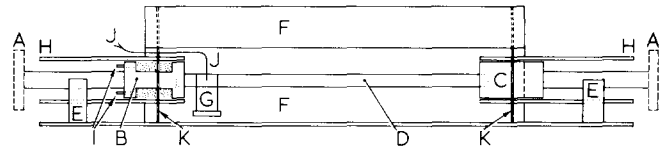

Figure 1 Apparatus (½ scale): A, clamp faces; B, heater; C, end support; D, sample; E, brass supports; F, matt black screen (partial cylinder); G, cotton markers and lead weight; H, glass sleeves; I, heater terminals; J, thermocouple (40 swg); K, stiff cardboard supports for screen

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A chrome–alumel thermocouple was sited at the heater face, trapped between the end of the rod and the heater by inserting it into a small slit provided for the purpose. In addition the rod was held firm against the face of the heater by clamping the whole of the free section of the apparatus (i.e. heater, rod and cold support) between the faces of a woodworker's clamp, thus ensuring good thermal contact. Two fine grooves were scribed around the test-rod at precisely 10 mm distance from each other, that nearest the heater being 5 mm from the heater face. By hanging loops of very fine black cotton over these grooves (weighted to hang vertically by means of a small lead weight) the positions of the markers were easily visible on the screen of the measuring instrument used and thus could be used for distance calibration. In addition a second chrome–alumel thermocouple of very low thermal mass was attached half way between the markers at a distance of 10 mm from the heater face, by means of which a temperature calibration point was obtained, the temperature at this point and at the heater face being measured using a sensitive electronic thermometer attached to the thermocouples. In addition the background (room) temperature was measured by means of a third thermocouple placed on the back surface of the surrounding paper screen at a point opposite the hot end of the rod.

Once the test piece was in position the heater was heated to a known temperature and the whole system allowed to come to equilibrium, a process taking not less than two hours. The thermal imaging system (see Appendix) was then calibrated by adjusting a suitable isotherm as displayed on the screen at the 10 mm mark. The known temperature of this isotherm could then be used to calculate the temperatures indicated by other isotherms displayed on any given instrument range. A millimetric scale applied to the face of the display screen of the instrument was used to determine the position of each isotherm with reference to the calibration provided by the cotton markers. As used the instrument provides a magnification of approximately 1.5, thus improving measurement accuracy. In this way a series of readings of the temperature of the test rod at varying distances from the heater could be obtained.

Having obtained these results, a short length of rod of similar material to that of the test-rod and having the same diameter was heated in a low-temperature oven to about 80°C. A fine chrome–alumel thermocouple was inserted into a 1 mm diameter hole approximately equidistant from the ends of the rod and drilled along the diameter of the rod to a depth slightly exceeding the rod's radius. When the rod was adjudged to have reached a uniform temperature throughout it was enclosed in an insulating sheath of expanded polystyrene and removed from the oven. It was then placed in a position in the apparatus similar to that occupied by the rod used in the earlier experiments, the heater of the apparatus being switched off and at room temperature. The polystyrene protection was then removed and the subsequent cooling of the rod measured by means of the thermocouple at half minute intervals, the time being measured by a stop-clock.

Results

The results for the steady-state distribution of temperature along a 10 mm diameter rod of PMMA are shown in *Figure 2*, the results obtained from three

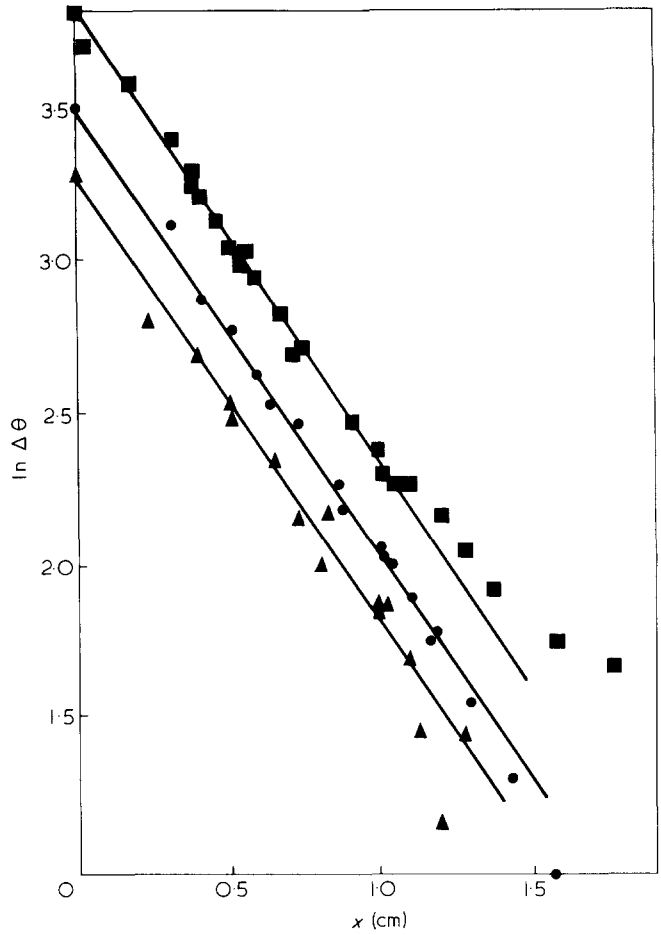

Figure 2 ln θ *versus* x: Heater face temperature: ▲, 49.8°C; $\partial(\ln \Delta\theta)/\partial x = -1.476$ cm^{-1}, significance coefficient = 0.9799, thermal diffusivity (k) = 1.53 x 10^{-3} cm^2 s^{-1}; ●, 59.35°C; $\partial(\ln \Delta\theta)/\partial x = -1.481$ cm^{-1}, significance coefficient = 0.9958, thermal diffusivity (k) = 1.55 x 10^{-3} cm^2 s^{-1}. ■, 67.1°C; $\partial(\ln \Delta\theta)/\partial t = -1.448$ cm^{-1}, significance coefficient = 0.9945, thermal diffusivity (k) = 1.62 x 10^{-3} cm^2 s^{-1}

experiments at different heater temperatures being given. *Figure 2* displays these results as a plot of ln $\Delta\theta$ *versus* distance where $\Delta\theta$ = measured temperature − background temperature (arbitrary zero).

Figure 3 shows the cooling curve for PMMA in terms of $\Delta\theta$ *versus* time and also of ln $\Delta\theta$ *versus* time.

Theory

The equation describing the temperature distribution along a thin bar of uniform cross-section and of semi-infinite length heated at $x=0$ to a constant temperature θ_{max} is:

$$\frac{K}{\rho c}\cdot\frac{\partial^2\theta}{\partial x^2}-\frac{Hp}{\rho cA}(\theta-\theta_0)=\frac{\partial\theta}{\partial t}$$

where H = outer conductivity, K = thermal conductivity, A = area of cross section of rod, p = circumference of rod, ρ = density of material, c = specific heat of material and θ_0 = background (room) temperature (taken as zero of temperature for the purposes of solution of the equation).

The steady-state (Forbes') condition gives:

$$\frac{K}{\rho c}\frac{\partial^2\theta}{\partial x^2}-\frac{Hp}{\rho cA}\Delta\theta=0$$

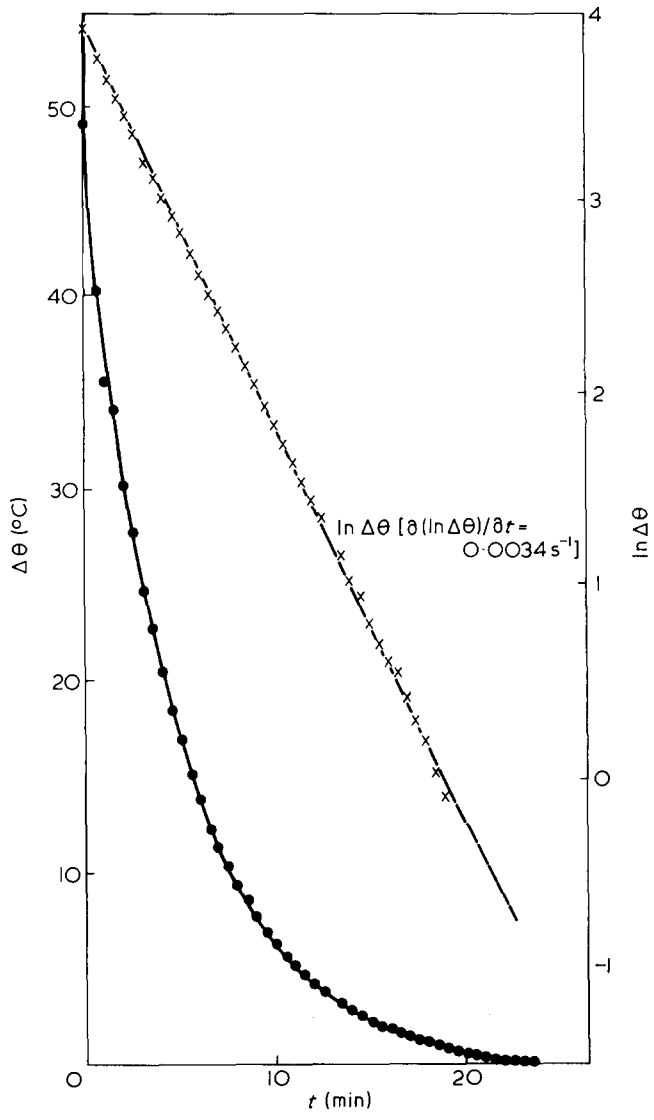

Figure 3 Cooling curve: $\Delta\theta$ *versus* time and ln $\Delta\theta$ *versus* time

and $\Delta\theta = \Delta\theta_1$ at $x = 0$ $\Delta\theta = 0$ at $x = \infty$.

This solves to

$$\Delta\theta = \Delta\theta_1 \exp\left(\frac{v}{\sqrt{k}}x\right)$$

where $v = \dfrac{Hp}{\rho cA}$ and k = thermal diffusivity $= K/\rho c$.

Forbes' method of measurement of thermal conductivity does not, however, rely on the application of this theoretical solution. Instead the method depends on simpler, more fundamental theory. At any point x on the bar the flux of heat across the cross-section is given by:

$$-KA\frac{\partial\theta}{\partial x}$$

Since there is no dissipation of heat from the surface of the rod at $x = \infty$, when $\Delta\theta = 0$, the heat transport by solid conduction at x must be equal to the heat lost from the surface of the bar in the region x to ∞.

The heat lost in time dt from an element of length $dx = A\,dx\,\rho c\dfrac{\partial\theta}{\partial t}dt$, from which it is easily shown that:

$$k\frac{\partial\theta}{\partial x} = \int_x^\infty \frac{\partial\theta}{\partial t}\cdot dx$$

Clearly $\dfrac{\partial\theta}{\partial x}$ at any x may be obtained from the distribution of temperature along the bar in the steady-state condition. The value of $\dfrac{\partial\theta}{\partial t}$ at any given temperature may be obtained from the cooling curve experimentally determined and thus for a given set of experimental results a curve of $\dfrac{\partial\theta}{\partial t}$ *versus* x may be constructed. The area under this curve from any given value of x to ∞ may thus be determined graphically and equated to $k\dfrac{\partial\theta}{\partial x}$, from which equation k, and therefore K, is readily calculated for the temperature selected.

Discussion

Figure 2 indicates that within the accuracy of the experiments the value of $\dfrac{\partial(\ln\Delta\theta)}{\partial x}$ is constant for PMMA in the temperature range studied. There is some apparent deviation from this relationship at low values of $\Delta\theta$ (less than ~ 5°C) but it is also true that errors of measurement of θ in this region are greater than at higher temperatures due to the broadening of the isotherms displayed by the measuring instrument in regions of low thermal gradient. It may be that these deviations are truly significant but in general results indicate that the slope $\dfrac{\partial(\ln\theta)}{\partial x}$ is substantially constant in the temperature range 30°C to 70°C, at least. Linear regression lines for the experimental results in this range give values of $\dfrac{\partial(\ln\theta)}{\partial x}$. The intercepts at $x = 0$ are within about 1°C of the values measured by thermocouple at the heater face and the significance levels of the linear regression analyses are in all cases very close to one.

The cooling curve shown in *Figure 3* is also seen to obey a simple logarithmic relationship of $\Delta\theta$ *versus* time to a very high degree of accuracy. Thus it is possible to write:

$$\frac{\partial\theta}{\partial t} = a\,\Delta\theta$$

where a is a constant over the range studied.

Combining the two logarithmic relationships so obtained it is clear that the value of the thermal diffusivity, k, is substantially constant over the relevant temperature range within the limits of accuracy of these experiments and:

$$k = \frac{a}{m^2} \quad \text{where} \quad \frac{\partial(\ln\theta)}{\partial x} = m.$$

The values of k obtained from the experiments are seen to be in reasonable agreement with each other. The

thermal conductivity K is readily obtained from these values by multiplying by the value of ρc obtained from the literature[7]. The variation of ρc with temperature over the range studied is small and K is found to increase by about 10% from room temperature to 70°C.

Published values of thermal conductivity for plastics are, in general, sparse and whilst a considerable amount of work has been done on PMMA below 100 K in this connection (for instance see Burgess and Greig[8]) values at room temperature and above are few. Eiermann[9] (also quoted by Choy[10]) gives a value of 4.8×10^{-4} cals cm^{-1} s^{-1} K^{-1} for the thermal conductivity of PMMA at 300 K. A paper by Knappe[13], also quoting Eiermann (see *Figure 10a*) gives a value of 4.5×10^{-4} cals cm^{-1} s^{-1} K^{-1} at 300 K and 4.58 cals cm^{-1} s^{-1} K^{-1} at 348 K. *Figure 12* in the same paper indicates two experimental values from Eiermann[11,12] of 4.6×10^{-4} and 4.8×10^{-4} cals cm^{-1} s^{-1} K^{-1} at 300 K. These values may be compared with those derived from the mean value of 1.57×10^{-3} cm s^{-1} for the thermal diffusivity of PMMA obtained from the three experiments reported in this paper and which leads to values of 5.7×10^{-4} and 6.3×10^{-4} cals cm^{-1} s^{-1} K^{-1} for the thermal conductivity at 300 K and 348 K respectively, using values of ρc from ref. 7. Reference 13 (*Figures 32, 33* and *Table 4*) and Choy[10] quote Eiermann and Hellwege[14] who reported an increase in thermal conductivity on stretched PMMA, measured in the direction of stretch. For a 375% extension a value of 6.64×10^{-4} cals cm^{-1} s^{-1} K^{-1} at 300 K is given. Since the sample used in the experiments reported here was extruded rod, some degree of orientation of the sample is certain, and from the results of ref. 14 the value of 5.7×10^{-4} cals cm^{-1} s^{-1} K^{-1} corresponds to 100–160% equivalent extension. The values obtained from this study are therefore seen to lie well within the expected range when the effect of orientation is taken into account.

Conclusions

Forbes' method of measurement of thermal conductivity is seen to give satisfactory results in the case of poly(methylmethacrylate), using modern infra-red temperature measuring equipment to measure the temperature gradient. It is reasonable to assume that the method would be equally applicable to other plastics and to solids of low thermal conductivity in general, such as glasses and ceramics. Greater accuracy would be expected with simple refinements of the apparatus, such as improved temperature control of the heater and enclosure of the apparatus in a well-controlled environmental chamber.

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Appendix

The aga thermovision. The instrument basically consists of two units, a camera and a display unit.

Infra-red radiation emitted by the body viewed is collected by a system of infra-red optics incorporating a large lens of pure silicon. The radiation is focussed on to the face of an indium antimonide photocell cooled by liquid nitrogen. The object is scanned by means of a mechanical scanning device so that radiation from small areas of the object viewed falls on the detector, the signal from which represents a measure of the intensity of radiation emitted by the area viewed at that particular moment. This signal is amplified and displayed on a television-type monitor whose raster of sixteen lines per second is synchronized with the mechanical scanning system. The intensity of the signal displayed on the cathode-ray tube at any given instant is related to the temperature of the small section of the object viewed and corresponding to that point on the screen. Thus a picture is built up on the screen in which the contrast is a representation of the temperature gradients at the surface of the object viewed. The sensitivity of the device may be adjusted by means of a fine control and a series of ranges determine the difference between the upper and lower limits of temperature displayed on the screen, the ranges being: 100°C, 50°C, 20°C, 10°C, 5°C and 1°C. In the experiments described only the four upper ranges were used. A scale beneath the display, after calibration, enables the temperature range in a given region of uniform intensity on the display to be measured. A succession of isotherms may also be displayed and the temperature corresponding to each may be determined after calibration, and it was this facility which was used in the experiments described in this communication. Further, a camera attachment may be used to photograph the screen and the isotherms displayed, if required.