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A PC-Based Transient Method for Thermal Conductivity Measurement

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ABSTRACT

In this paper, an indigenously developed thermal probe has been interfaced with a PC for automated measurement of thermal conductivity (K). The developed system has been calibrated and standardised by measuring K of glycerol. The maximum percentage error, for repeated sets of observations, was within ± 7.29 per cent of standard value reported for glycerol. This methodology has been successfully employed for measuring K of propellant oxidisers, additives, binders, etc.

NOMENCLATURE

E_i	Exponential integral
r	Distance between heat source and sensor
T	Temperature
t	Time
T_1	Temperature at time t_1
T_2	Temperature at time t_2
q	Heat per unit source length
α	Thermal diffusivity
γ	Euler's constant

1. INTRODUCTION

The knowledge of effective thermal conductivity (K) of a composite medium or a multiphase system is of importance for a wide range of engineering problems. For instance, the effective K determines the temperature field in an explosive, which in-turn determines the detonative response of the explosive[1,2]. A violent, typically detonative response of explosives to heat, has been a major cause of damage/accidents. Two heating rate regimes are of concern in munitions technology, commonly referred to as fast and slow cook off conditions. A fast rate of heating results from direct exposure to fuel fire and leads to large radial temperature gradients across the explosives, and ignition, at or close to the surface of the explosives. In contrast, a very slow rate of heating is due to prolonged indirect exposure to a heat source and leads to thermal equilibrium condition across the explosives. The nature of thermal response of these materials depends upon the thermal properties of the explosive composites amongst other factors. The cook off response of explosives is also influenced by the extent of coating of the polymeric binder on the explosive[3], and can be moderated by blending it with more thermally stable and less sensitive explosives. Dagley[4-5], *et al.* have shown the importance of K when modelling heat flow in explosives. The tests are typically carried out at both fast (Approx 1 °C/s) and slow (Approx. 0.1 °C/s) heating rates.

In these models[4-6], K of explosives has been calculated from the expression derived by

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Maxwell[7], and does not include the effect of particle size, shape, temperature, porosity, etc. However, these are the main factors affecting the properties of K. These conductivity expressions only provide a general idea and do not give the exact value, which plays a major role in assessing the magnitude of temperature within the material. In particular, when one works with explosives of various types, reliable data of K is only obtainable through experimentation. There are various expressions[8] available in the literature for predicting the effective value of K of porous and dispersed systems, but none of these can replace the experimentally determined value of K.

There are steadystate and transient methods for the measurement of K of the materials. The results for single-phase materials using these two methods are generally in good agreement. Though steadystate methods are simple theoretically, but involve rather elaborate technique practically, including thermal guard system to eliminate lateral heat flow and electronic control system to ensure stable conditions during the test. The system is often provided with complex heat guards to maintain a temperature profile similar to the temperature of the sample and requires a large quantity of test sample for such measurements[9-11]. Moreover, these methods don't permit *in situ* measurements. The transient technique provides fast measurement, saving time, and a distinct advantage being that it can be used for *in situ* measurements. The method reduces unwanted modes of heat transfer as less time is required for taking the observations. The techniques are extensively used for the determination of K of different types of materials like alloys, semiconductors, ceramics, dispersed and layered composites, soils, porous and granular materials[12,13], etc.

In the present study, the transient method called thermal probe method has been used for the determination of K. The thermal probes used have been indigenously developed, calibrated, and standardised in the laboratory; and the measurement system has been computerised. Propellant oxidisers, additives and binders have been used for the measurements of K. The main emphasis of the study is on the automation of the measurement system.

2. BASIC MEASUREMENT PHILOSOPHY

The thermal probe method is based on the simplified mathematical solution of an infinite line heat source in a homogeneous medium[14,15]. Thermal probe is made of metal cylinder with integral constant power heater and temperature sensor (Fig.1) which after being buried in the sample is allowed to come to thermal equilibrium and then the power is switched on. The temperature of the cylinder (probe) then rises as heat dissipates. The rate of heat dissipation is a function of the thermal conductance of material, besides other factors. The values of K can be evaluated using a suitable identification procedure and recording the variation of probe temperature with time.

The present method for analysing available temperature data has been derived from the theory of heat conduction of a line heat source embedded in an infinite homogeneous medium. The theoretical solution of the problem shows that the temperature response of the probe, when plotted against the natural logarithm of time, becomes linear at relatively longer time.

2.1 Theory of Probe Method

Taking a thermal probe as an infinite line heat source in an infinite homogeneous medium, the temperature response[16] at any radius, r, is given by

$$T(r) = -\frac{q}{\quad} E_i\left(\frac{r^2}{4\alpha t}\right) \tag{1}$$

Equation (1) can be expanded as

$$T(t) = \frac{q}{4\pi K}\left[\ln\left(\frac{4\alpha t}{r^2}\right) - \ln\gamma + \frac{r^2}{4\alpha t} - \frac{1}{4}\left(\frac{r^2}{4\alpha t}\right)^2 + \ldots\right] \tag{2}$$

For long time, only the first two terms are significant and the temperature rise at any point may be approximated as

$$\frac{q}{4\pi K}\left[\ln\left(\frac{4\alpha t}{r^2}\right) - \ln\gamma\right] \quad (3)$$

or

$$\frac{q}{4\pi K}\left[\ln(t) + \ln\frac{4\alpha}{r^2\gamma}\right] \quad (4)$$

It is apparent from Eqn (4) that if the temperature at the source, or at any radial distance from it, is plotted as a function of $\ln(t)$, the slope is $q/4\pi K$, from which K can be calculated using Eqn (5) provided q supplied to the probe is known.

$$T - T_1 = \frac{q}{} \ln\left(\frac{t_2}{t_1}\right. \quad (5)$$

The value of q is calculated by measuring the current in probe heater wire of known resistance.

2.2 Design of Thermal Conductivity Probe

It is desirable that the time of measurement should not exceed 5-6 min and that the temperature rise of the probe and the adjacent medium should not be more than 13-14 °C for dry medium and 8-10 °C for moist medium. It will reduce moisture migration if any, and also the rise in temperature of the sample will not be significant. For accuracy and data reduction, it is desirable to use a probe which approaches, in a thermal sense, the limiting case of line heat source, by increasing length-to-diameter ratio (= 100) and decreasing thermal mass. Using the theory of line heat source, the behaviour of practical thermal probe can be analysed and its thermal response can be described by the simple line heat source equation, without introducing appreciable error in the results.

Considering the above factors, probes are designed and constructed. A thread-insulated heater wire of constantan (36 gauge) is stretched double or triple-fold centrally along the hollow stainless steel needle. An insulation paint-enamelled copper-constantan (T-type) thermocouple (TC) of same gauge is placed in the needle up to the mid point of the probe for measuring mean temperature variations. Heavy copper leads are soldered to the constantan heater wire terminals. TC and heater leads are then brought out through a plastic cap. The other end of the needle is sealed with a brass or steel conical plug, facilitating an easy insertion of the probe in test material. To fix the position of wires in the cylindrical envelope, to remove air and to prevent electrical contact, silica powder (insulator) is used. Another advantage of filling the probe with insulator is to minimise the convection, and so conduction is the main mode of heat transfer.

TC that drifts excessively due to heat, air current (environmental conditions), produces inconsistent results. In a laboratory, a properly maintained ice bath gives the consistent reference temperature for TC. In practice, the major deviation from the theoretical probe response is due to the thermal contact between the probe and the sample. If the probe has no heat capacity, it would cause only an instantaneous offset in the temperature when probe power is initiated. Because of probe heat capacity, the contact related temperature rise gradually approaches the offset value. Whereas the effect of probe heat capacity is predictable, the contact conductance depends on the material under investigation and the skill of the operator in installing the probe with minimum disturbance. The diameter of thermal probe can affect the magnitude of the thermal contact resistance. Poor probe-to-sample contact results in a longer time to approach the straight line portion of the curve, i.e., initial transient time, but does not change the absolute value of K which is derived from the slope of the straight line.

The theory for the transient K-test is based on an infinite sample size. Therefore, if a sample container of given radius is used, boundary effects are evident after a certain time. The actual test results must be separated from these effects. The time after which the boundary effect is evident, can be estimated from

$$Exp\left(r/4\alpha t\right) << T$$

In practice, this approach gives the time when temperature first impinges on the boundary. It takes

long time for these boundary effects to cause a deviation from normal thermal probe behaviour. By placing a *TC* at the sample container boundary, boundary effects may be observed.

The amount of input power to the probe, for the tested sample, must be carefully selected to ensure that conduction is the only mode of heat transfer. A graph between temperature measured from sensor and natural logarithm of time gives the value of K of material in which the probe is placed. The experiment involves obtaining the values from a graphical plot and subsequently performing computations, and the computerisation of the experiment would provide the user with the graphical data as well as the results (i.e., K of the material).

3. DATA ACQUISITION SYSTEM DESIGN

The computerisation involves the interfacing of probe to a PC and accomplishing the desired functionality in an application program as shown in Fig. 1. The steps involved in the process are: selection of required hardware, interfacing and data acquisition, and writing application program. The flow chart for the application program is shown in Fig. 2.

3.1 Hardware Requirements

The thermal probe, which is used to measure the temperature changes in the material, has a built-in TC. The thermo-emf thus generated is proportional to the temperature difference between the two junctions. As the output of TC is of the order of a few mV, it needs to be amplified to be measured by the data acquisition card. This is achieved by the use of commercially available precision instrumentation amplifier, viz., AD521JD. The AD 521 is a precision differential voltage[17] gain device optimised for operation in a real world environment and is intended to be used whenever acquisition of useful signal is difficult. As a complete instrumentation amplifier, the AD521 requires only two resistors to set its gain to any value between 0.1 and 1000. It is characterised by high input impedance of the order of 3000 MΩ, high common mode rejection ratio (CMRR), low bias currents and low drift. The output of the

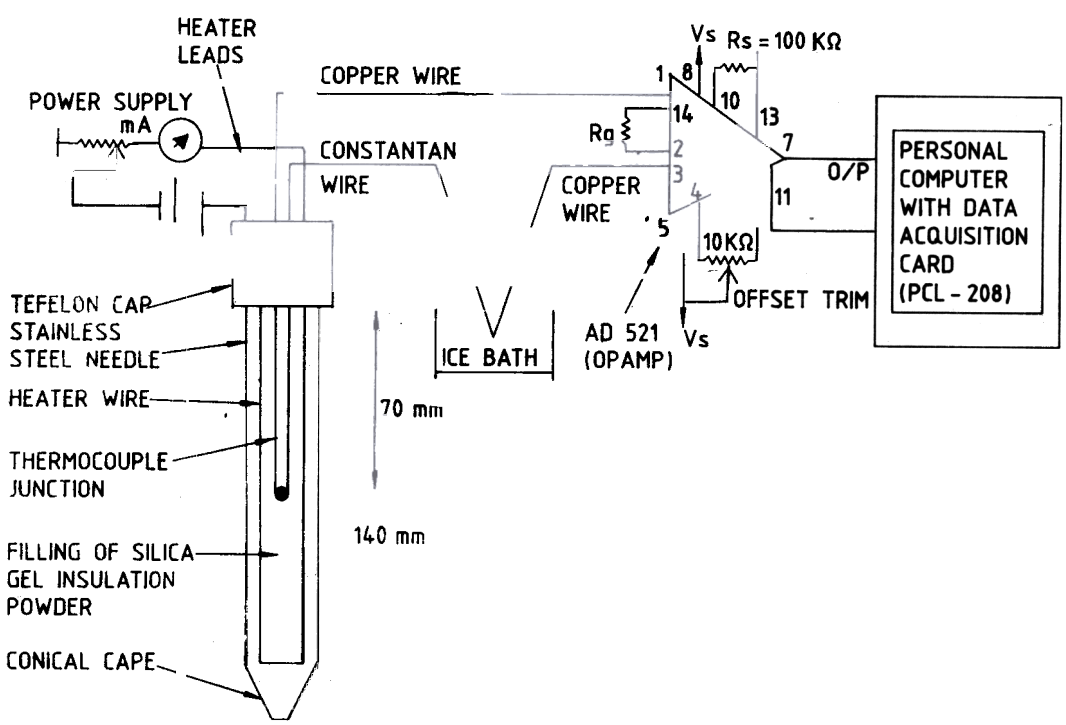

Figure 1. Electronic diagram of thermal conductivity measurement system

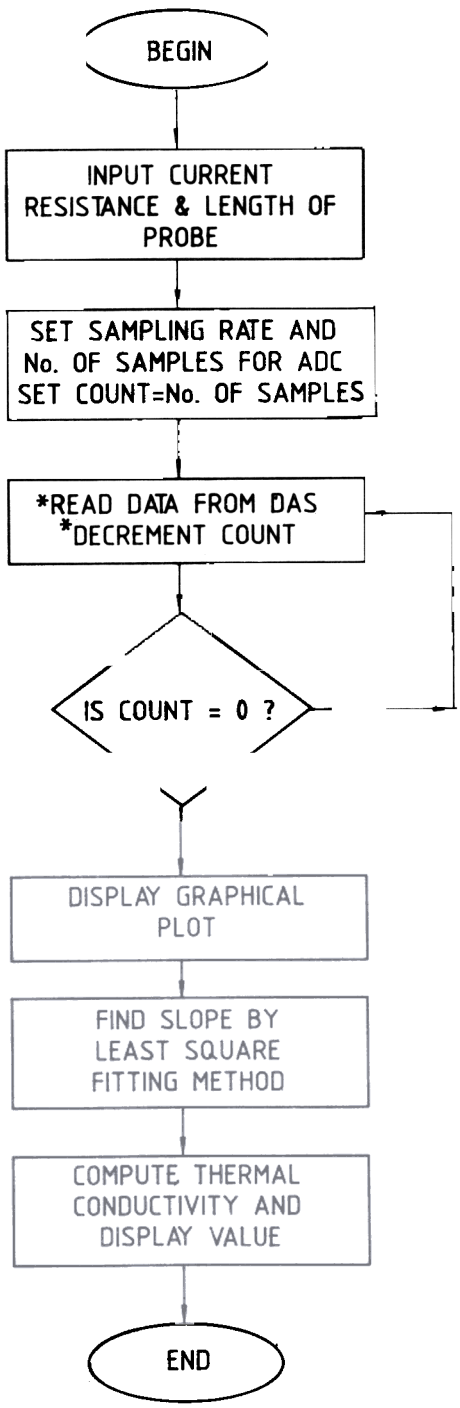

Figure 2. Flow chart for PC-based thermal conductivity measurement system.

instrumentation amplifier is fed to the analog data input of the A/D converter and the digital data is read by the host PC. A software trigger is used to read successive data at desired time intervals.

3.2 Data Acquisition System

The data acquisition system consists of a data acquisition card (PCL208) which is interfaced to the PC. It is a high performance, high speed, multifunction data acquisition card for the IBM PC/XT/AT or compatibles. The key features of this interface control card are:

- Switch selectable 16-single ended or 8-differential analog input channels

  An industrial standard 12-bit successive approximation A/D converter with a maximum sampling rate of 60 KHz in DMA mode

  Switch selectable versatile analog input ranges

  Three A/D trigger modes: Software trigger, programmable pacer trigger and external pulse trigger

- An INTEL 8254 programmable timer/counter provides pacer output (trigger pulse) at the rate of 2.5 MHz to 71 min/pulse to the A/D. The timer time base is switch selectable for 10 MHz or 1 MHz.

3.3 Interfacing & Data Acquisition

The next step is the interfacing of the data acquisition card to the PC. This is achieved by writing driver routines in a suitable programming language. The obvious choice of programming language was C due to its versatility. The data acquisition system performs various functions which include applications of A/D, D/A, digital I/O and programmable interval timer. It also covers different types of A/D applications like A/D with interrupt transfer, DMA transfer, etc. This driver routine performs the following functions:

- Checks the hardware

  Initialises the card by writing the appropriate control word in the control register of the PCL208 card

- Provides software trigger

- Allows the user to set the channels to be monitored for data acquisition

Checks on the number of input channels to be used and whether the signal is single or differential.

Most of the PC peripheral devices and interface cards are controlled through the I/O ports. These ports are addressed using the I/O port address space. I/O port base address for the PCL208 is selectable using a 8-position DIP switch. PCL208 requires 16-consecutive address locations in the I/O space. Thus, based on the register structure, data format and hardware of the card, a program is written in C for A/D conversion and data acquisition.

3.4 Application Programs for Computation & Graphical Display

The last step is writing application programs to determine K of the material. These programs written in C, acquire the data, perform the required computations and calculate K of the material. A graphical plot of the data obtained is also made available, and from this graph, the slope is determined which is used for the computation of K of the material. The program also has the provision to determine the best fit for the line (using least square method).

4. RESULTS & DISCUSSION

As a preliminary step to standardise the probe, calibration measurements are performed. The material chosen for the purpose is glycerol (of BDH make and 99.9 per cent purity). Glycerol was chosen as a suitable material because contact resistance errors can be minimised for probe to liquid contact. Furthermore, since glycerol is viscous, convective heat transfer effects are reduced. Current is selected, such that overall temperature rise is below 13-14 °C. A specimen plot of probe temperature rise *versus* logarithm of time for glycerol is shown in Fig. 3. A number of measurements for glycerol are taken and these values are presented in Table 1. The measured values of K for glycerol are in agreement with the values reported in the literature, with maximum observed error of 7.29 per cent for the above set of

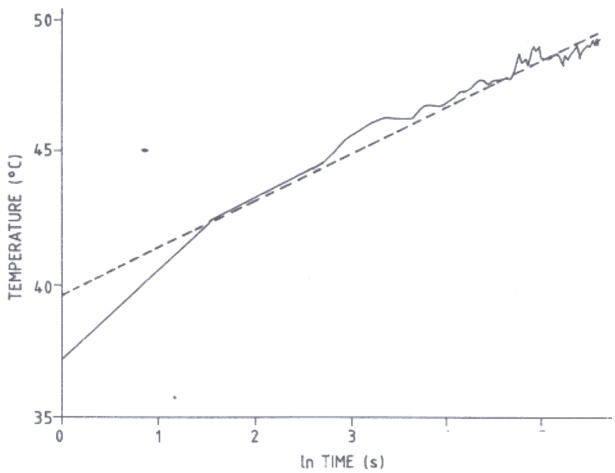

Figure 3. Graphical PC output of temperature with time on logarithmic scale and straight line curve fitting for glycerol.

measurements. The standard deviation calculated is 0.014 and presented in Table 1.

In a similar manner, the values of K of RDX, HTPB, CAB, DOA, aluminium powder and di-nitroso pentamethylene tetra-amine (DNPT) are also measured. For each sample, five tests are conducted and the standard deviations are also

Table 1. Measured values of thermal conductivity of glycerol (99.9 per cent pure) and percentage error analysis

Measured value of thermal conductivity (W/mk)	Percentage error (0.289 W/mk standard value of glycerol)	Standard deviation
0.269	– 6.92	0.014
0.280	– 3.22	
0.278	– 3.80	
0.280	– 3.11	
0.290	0.34	
0.300	3.80	
0.290	0.34	
0.300	3.80	
0.290	0.34	
0.310	7.26	
0.290	0.34	
0.290	0.34	
0.310	7.26	
0.280	– 3.11	
0.269	– 6.92	

Table 2. Measured values of propellant oxidisers, additives and binders at ambient temperature

Test No.	Sample	Thermal conductivity (W/mk)	Standard deviation	Reported thermal conductivity (W/mk)
	RDX	0.31	0.012	0.26
ii		0.30		
		0.28		0.27
iv		0.29		
		0.30		
	HTPB	0.15	0.01	0.20
ii		0.17		
iii		0.14		
iv		0.16		
		0.16		
	DNPT	0.31	0.015	
		0.33		
iii		0.34		
iv		0.35		
v		0.33		
	CAB	0.47	0.021	
ii		0.48		
iii		0.50		
		0.46		
		0.51		
	Aluminium powder	0.20	0.009	
		0.22		
		0.22		
		0.21		
v		0.22		
	DOA	0.11	0.006	
ii		0.10		
		0.09		
		0.09		
		0.10		

calculated and presented in Table 2. The values of K reported for RDX and HTPB[1,4,19] are also shown in Table 2 and agree closely with the measured values of K. The slight deviation from the reported values may be due to the fact that the values of K of the material depend upon physical properties, i.e., density, particle size, porosity, temperature, grain-type and moisture content, if any, besides K of solid constituent. Different methods of measurement, i.e., transient or steadystate, may also be the possible cause of small deviations.

5. CONCLUSION

The probe method is suitable for the measurement of effective values of K of dry and moist, loose and granular materials. This method can also be used for the measurement of effective values of K of liquids. The developed instrumentation is quite accurate. The apparatus is feasible, nondestructive and simple to use. This method gives good results for effective values of K of porous materials as compared to steadystate methods. In addition, probe method is based on a simplified mathematical solution for the case of an infinite line heat source in a homogeneous medium with constant thermal properties.

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