A CYCLIC HEAT FLOW METER APPARATUS TO MEASURE THERMAL CONDUCTIVITY AND/OR DIFFUSIVITY

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A CYCLIC HEAT FLOW METER APPARATUS TO MEASURE THERMAL CONDUCTIVITY AND/OR DIFFUSIVITY

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ABSTRACT

A cyclic heat meter apparatus has been developed for measuring thermal diffusivities or thermal conductivity and diffusivity of flat slab specimens. The specimens may be moist or dry. The assumption that a moist soil may be considered a normal heat conducting solid has been validated for a moist clay. The reproducibility of the apparatus is about 3 per cent.

On a mis au point une méthode servant à mesurer les diffusivités thermiques ou la conductivité et la diffusivité thermiques d'échantillons sous forme de plaques à l'aide d'un fluxmètre thermique en régime cyclique stabilisé. Les échantillons peuvent être humides ou secs. L'hypothèse selon laquelle un sol humide peut être considéré comme un conducteur normal de la chaleur est confirmée. La méthode a une reproduisibilité d'environ 3 pour cent.
Introduction

When a temperature gradient exists through a porous material which contains some moisture, heat is transferred by two mechanisms. There is the normal thermal conduction and a parallel heat transfer due to the migration of the moisture toward the region of lowest temperature.

Studies of the annual variations of ground temperature suggest that the ground can be represented as a normal heat conducting solid whose apparent thermal diffusivity and conductivity are functions of the soil type, density, and mean moisture content. This raises the problem of how to determine the appropriate thermal conductivity and diffusivity of moist soils and other porous materials. Kirkham and Jackson (1) and Higashi (2) have reported methods that use periodic (cyclic) boundary conditions to determine the diffusivity of moist samples. In 1959 the Division of Building Research developed a laboratory apparatus similar in principle to these but measuring thermal conductivity as well as diffusivity. This paper describes the current form of the DBR apparatus and method and discusses the accuracy of the results for a neoprene rubber slab and a moist clay soil.

The Cyclic Heat Meter

To maintain a constant mean temperature and mean moisture content through a sample during test it is necessary to use a temperature gradient that is periodically reversed. The Cyclic Heat Meter used at DBR consists of two 30- by 30-cm aluminum heat exchanger plates which can be kept at a controlled temperature by circulating controlled temperature liquid through them. The liquid is supplied by two circulating laboratory baths each of which can be controlled at any temperature between -70C and 90C.
The baths and plates are connected to a valve matrix as shown in Fig. 1. A two circuit patch board allows one set of valves to be energized while the other is de-energized. Powering of the groups can be reversed by activating a remote switch. A computer acts as a cycle timer, closing the switch for 50 per cent of the period. The baths are set at different temperatures. The resulting temperatures on the surface of the specimens are equal amplitude near-square waves, once initial transients disappear. The waves can be either in-phase or 180° out-of-phase as selected by the programmer.

Two slabs of materials are placed between the heat exchanger plates (Fig. 1). Temperatures are measured at planes 1, 2 and 3 with 36-gauge butt-welded thermocouples. Three couples are used at each plane; one at the centre of the area and one 5 cm to each side of centre. When the couples indicate the same temperature it shows that the heat flow in this central region is one dimensional. The requirement for one-dimensional heat flow limits
the total thicknesses of specimen and standard slab to about 8 cm. When the properties of one slab of material are known and those of the other unknown, the configuration is identical to a heat flow meter apparatus. Because in this case the plate temperatures can be cycled, the apparatus is termed the cyclic heat meter.

Data Acquisition and Control System

A computer-based, data acquisition and control system controls the apparatus, acts as the cycle timer, collects, preprocesses, displays and records the data. A programmable gain, 15-bit analogue to digital converter measures the filtered output from the nine thermocouples in 9 milliseconds at intervals of 0.1 second. The data are filtered digitally with a single-stage binary filter, then recorded at a selected interval on magnetic tape. An off-line computer is used for final processing. A simpler system, used successfully during part of the study, was based on a multi-point strip chart recorder with punched paper tape output.

Periodic Heat Flow in a Composite Slab

For a homogeneous slab with sinusoidal temperature variation at both surfaces, the surface temperatures and heat flows are related by linear equations which may be expressed conveniently in the matrix notation (3). For the situation shown in Fig. 1 this is

\[
\begin{align*}
\theta_1 & = A_1, B_1 \\
\theta_2 & = A_2, B_2 \\
\theta_3 & = A_3, B_3
\end{align*}
\]

and

\[
\begin{align*}
q_1 & = C_1, A_1 \\
q_2 & = C_2, A_2 \\
q_3 & = C_3, A_3
\end{align*}
\]

where: \( \theta_j \), \( q_j \) = harmonic components of temperature and heat flow with period \( t_0 \) at position \( j \). The matrix elements for slab \( k \) are:

\[
\begin{align*}
A_k & = \cosh (lt) \phi_k \\
B_k & = (R_k/(1+i)\omega_k) \cdot \sinh (1+i)\omega_k \\
C_k & = ((1+i)\omega_k/R_k) \cdot \sinh (1+i)\omega_k; \quad R_k = d_k/\lambda_k, \text{ thermal resistance; } \\
d_k & = \text{thickness; } \lambda_k = \text{thermal conductivity; } \omega_k = (ld_k^2/D_k t_0)^{1/2},
\end{align*}
\]
nondimensional frequency; \( D_k = \text{thermal diffusivity} = \frac{\lambda_k}{\rho_k c_k} \); \( c_k = \text{heat capacity}; \rho_k = \text{density}. \)

If the periodic driving temperature is not a simple sinusoidal variation it can be expressed as the sum of a series of components, each a sine wave, with periods that are simple fractions of the fundamental period. When \( \lambda \) and \( D \) are not temperature dependent, the effect of each component can be calculated as if it alone was present and the effects added to give the total effect. When the materials are slightly temperature dependent, as they usually are, the fundamental component of temperature inside the slab is almost unaffected (4, 5). The higher harmonic components are changed in amplitude and phase and a mean temperature shift is generated in the material. The amplitude and phase of the fundamental component is masked by these effects. This is true even when pure sine waves are applied to the surfaces. An accurate Fourier Analysis or Fast-Fourier Transformation is necessary to recover the fundamental component. This is not practical without automatic data recording equipment and computer analysis.

**Determination of Properties from Harmonic Components**

Equations (1) and (2) can be rearranged to give

\[
\frac{A_1 - \theta_1/\theta_2}{B_1} + q_2/\theta_2 = 0 \quad (3)
\]

\[
q_2/\theta_2 = \frac{(A_2 - \theta_3/\theta_2)/B_2}{.} \quad (4)
\]

Combining these gives

\[
\frac{(A_1 - \theta_1/\theta_2)/B_1 + (A_3 - \theta_3/\theta_2)/B_2}{= 0} \quad (5)
\]

Equation (5) is a complex equation. It can be split into either modulus and argument or real and imaginary parts. The two equations can then be solved for any two unknowns. \( A_1, A_2, B_1 \) and \( B_2 \) contain the parameters \( R_1, R_2, \omega_1, \omega_2, L_1 \) and \( L_2 \). If four of these plus \( \theta_1, \theta_2 \) and \( \theta_3 \) are known, the remaining two can be determined.
Should equal contact resistances, $R_f'$, occur on each interface, and if the thermocouples measure a temperature at a mid-point in the resistance and the two slabs are identical, the equation equivalent to equation (5) is

\[
\frac{\theta_1 + \theta_3}{2} = 2 \cdot A + R_f' \cdot C/2
\]  

(6)

The most pertinent combinations of knowns and unknowns used in solving equations (5) and (6) are as follows:

<table>
<thead>
<tr>
<th>Given</th>
<th>Solve for</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. $\varphi_1, R_1$</td>
<td>$\varphi_2, R_2$</td>
</tr>
<tr>
<td>2. $R_1, R_2$</td>
<td>$\varphi_1, \varphi_2$</td>
</tr>
<tr>
<td>3. $L_1, L_2, \lambda_1 = \lambda_2$</td>
<td>$\varphi_1, \varphi_2$</td>
</tr>
<tr>
<td>4. materials identical</td>
<td>$\varphi, L_1/L_2$</td>
</tr>
<tr>
<td>5. materials identical, equal contact resistances</td>
<td>$\varphi, R_f$</td>
</tr>
<tr>
<td>6. materials identical, $L_1, L_2$</td>
<td>$\varphi$</td>
</tr>
</tbody>
</table>

Several procedures have been used for solving these simultaneous, non-linear equations. In case No. 6 the equations have been solved explicitly for $\varphi$. In cases Nos. 1, 2, and 5 the equations were divided into modulus and argument to yield one equation in $\varphi$ and the other for $R$ or $R_f$ in terms of $\varphi(6)$. The first equation has been solved for $\varphi$ graphically, or to a more satisfactory precision, numerically by the Regula Falsi (7), Newton-Raphson (7), and Mueller (7) methods. The value of $\varphi$ was then used to determine $R$ or $R_f$. All these numerical methods have been found to experience problems occasionally when singularities, caused by experimental errors, occurred near the solution. In certain cases even small experimental errors would preclude a solution. All cases have been solved using Powell's Method (7). This method worked whether or not the equations were divided into
modulus and argument or real and imaginary parts.

Powell's Method can either solve the simultaneous equations for the unknowns or, where no exact solution exists, find the set of values of the unknown that came closest to satisfying the equations. In the latter case, the sum of the least-square errors in the solutions is minimized. The process is termed "finite dimensional optimization." It requires an initial guess at the solution. This is obtained from one of the methods already mentioned or from a knowledge of the material's properties. The method converges very rapidly on the best least-squares solution. The solution obtained by this method has in many cases differed significantly from those obtained by the other methods. Powell's method has been used for case No. 6 to find the single value of $\phi$ that best satisfies two equations, again in the least squares sense.

Experimental Procedure

The experimental procedure is simple: the mean temperature and amplitude are set by adjusting the set points for the temperatures of the two baths and the frequency is set by entering the desired value into the computer. The phasing is selected manually. The start and termination of recording on tape is controlled by the computer. The tape is later processed on the off-line computer. The conversion of the readings to temperature and the Fourier Transformation are performed first. Then thermal properties are determined using one or more of the numerical methods described. At least five independent sets of answers are calculated for each test. The mean and standard deviation are then calculated. When desired, the results for other harmonics, principally the third and fifth, are calculated. In general only the first harmonic is used because of sensitivity of the higher harmonics to sources of errors. The total amount of computer time used varies
depending on the number of cycles processed, but is normally less than 15 minutes.

**Experimental Studies and Results**

An experimental program was carried out to check the validity of the assumptions used in the analysis of the results. The experiments can be divided into two series: tests with rubber slabs to prove that the method works for dry materials, and tests with moist Leda clay to prove that the method works for moist materials.

The neoprene rubber slabs were approximately 2.5 cm thick. Two pairs with differing density were selected. The thermal resistance of each set was determined with a 30-cm guarded hot plate apparatus to an accuracy of about $\frac{1}{2}$ per cent. The Leda clay slabs were prepared in the laboratory with a thickness of one inch and moisture content of 21 per cent. The maximum coupling of heat and mass transfer was predicted to occur near this moisture content.

In the first series of tests, the slabs of rubber were tested in pairs to determine $\omega$, then one of each pair was used to test the method of obtaining $R$ and $\phi$. In the second series of tests one slab of rubber was used with the slab of soil and the $R$ and $\phi$ of the soil were determined. In each series the amplitude and frequency of the temperature oscillations were varied. The per cent deviation from the mean of $D$ for both the rubber and soil and of $\lambda$ for the soil obtained in the tests are plotted in Fig. 2. Most of these data were obtained before the apparatus was improved. The reproducibility of the simpler form of the apparatus was not as good as the current version. In spite of this, there is little variation in either parameter. The thermal conductivity of the rubber slabs
determined from the cyclic heat meter results agreed with the guarded hot plate result to within $\frac{1}{2}$ per cent.

The accuracy of the diffusivity measurements could not be determined due to a lack of a suitable standard. A comparison with the measurements from a calorimeter indicated an agreement to within 3 per cent. This was less than the sum of the possible errors in the apparatuses.

A separate sensitivity analysis too long to report here indicated the reproducibility of the current apparatus to be better than 1 per cent with the type of specimens used in these tests.

Variation of D and $\lambda$ of rubber and soil with largest temperature on surfaces. (Data from current and former apparatuses.)
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References


