PEAK-TIME METHOD FOR MEASURING THERMAL DIFFUSIVITY OF SMALL SOLID SPECIMENS

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METHODE DE TEMPS DE POINTE POUR MESURER LA DIFFUSIVITE THERMIQUE DE PETITS SPECIMEN SOLIDES

SOMMAIRE

Une méthode d'essai simple est développée pour déterminer la diffusivité thermique des matériaux solides et granulaires en mesurant le temps auquel la pointe survient sur un record température temps. Elle permet l'usage de petits spécimens facilement préparés et des spécimens de forme irrégulière (i.e. pierres). Les essais peuvent être effectués à une température normale aussi bien qu'à des températures élevées, et peuvent être répétés à courtes intervalles.
Peak-Time Method for Measuring Thermal Diffusivity of Small Solid Specimens

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A simple test method is developed for determining the thermal diffusivity of solids and granular materials by measuring the time at which the peak on a temperature-time record occurs. It allows the use of small easily prepared specimens and specimens of irregular shape (for example, stones). The tests can be performed at room temperature as well as at high temperatures, and can be repeated in short time intervals.

The principal difficulties of measuring the thermal diffusivity of solids are associated with specimen preparation. Very often a specimen has to be machined or ground to a certain shape, and for solids of considerable hardness this operation may be extremely time-consuming and costly. The difficulties are further increased by the fact that it is often next to impossible to find a piece of the solid sufficiently large to serve as a specimen.

This latter problem is especially acute with some concrete aggregates that are often unavailable in sizes larger than about 1½ in. As the thermal conductivity of aggregates is known to be the primary factor in the conductivity of concrete (1, 2), inability to determine this property seriously hinders the design of concrete mixtures in applications where thermal characteristics are of principal importance.

The method to be described for measuring thermal diffusivity of solids is notable for its simplicity with regard to both specimen preparation and the performance of the test. It is especially suitable for nonmetallic materials, and can be carried out on very small specimens.

THEORY

It will be shown that a method developed earlier (3) for the measurement of thermal diffusivity, using idealized semi-infinite solids, can be extended to include the use of
solids of more convenient geometry, such as cube, sphere, and even to solids of irregular shape.

If heat is supplied for a short period to the surface of a solid body, initially in equilibrium with its surroundings, the temperature at any point inside the solid will exhibit a maximum at some time following cessation of the heat supply. The thermal diffusivity of the solid can be determined by measuring the time at which the maximum temperature occurs. (For this reason it seems appropriate to refer to this method as peak-time method.)

By generalization of the results in reference 3, one can assume that, at least approximately, the following relation is applicable to the peak

\[
\frac{\kappa t_m}{\ell^2} = A \Phi \left( \frac{\tau}{t_m} \right) \Phi \left( \frac{h \ell}{k} \right)
\]

where the coefficient \(A\) depends on the geometry of the solid body and on the location of the point of interest inside the body. As the \(\Phi\), and \(\Phi\) functions are such that

\[
\left\{ \begin{align*}
\Phi \left( \frac{\tau}{t_m} \right) & \to 1 & \text{as } \frac{\tau}{t_m} \to 0 \\
\Phi \left( \frac{h \ell}{k} \right) & \to 1 & \text{as } \frac{h \ell}{k} \to \infty
\end{align*} \right.
\]

the two functions are actually correction terms applicable when the \(\tau/t_m \to 0\) and \(h \ell/k \to \infty\) conditions cannot be approximated closely enough in the experiment. (Approximate expressions for \(\Phi\) and \(\Phi\) will be presented later.)

The coefficient \(A\) can be determined by examining the solution of the following heat conduction problem:

\[
\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} = \frac{1}{\kappa} \frac{\partial T}{\partial t} \quad \text{when } t > 0 \quad \text{in } V
\]

\[
T(x, y, z) = T_0 + 1 \quad \text{when } t > 0, x, y, z \text{ on } S \quad (8)
\]

on the condition that \(\tau \to 0\).

If \(T(x/l, y/l, z/l, \kappa t/l^2)\) represents the solution obtained when the above boundary conditions (Equations (6) and (7)) are replaced by

\[
T(x, y, z) = T_0 + 1 \quad \text{when } t > 0, x, y, z \text{ on } S
\]

then the solution to the problem stated by Equations (4) to (7) is obtained as

\[
T = (T_p - T_o) \left[ \frac{1}{T(x/l, y/l, z/l, \kappa t/l^2)} - \frac{1}{T(x/l, y/l, z/l, \kappa t/l^2)} \right] (9)
\]

or, in the limit as \(\tau \to 0\), as

\[
T = \frac{\tau}{T_p - T_o} \frac{\partial}{\partial t} T(x/l, y/l, z/l, \kappa t/l^2) \quad (10)
\]

Because at the maximum \(\partial T/\partial t = 0\), the time coordinate of the maximum temperature (in the limit as \(\tau \to 0\)) can be expressed from the equation

\[
\frac{\partial^2 T}{\partial t^2} (x/l, y/l, z/l, \kappa tM/l^2) = 0
\]

and the resulting expression is of the form

\[
\frac{\kappa tM}{\ell^2} = A(x/l, y/l, z/l)
\]

It is logical to select the most important point of the solid, its center of gravity, as the point at which the temperature variation is to be observed. With this choice \(A\) becomes a unique function of the shape of the body.

<table>
<thead>
<tr>
<th>Solid Body</th>
<th>A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sphere</td>
<td>0.0918</td>
</tr>
<tr>
<td>Infinite Circular Cylinder</td>
<td>0.1170</td>
</tr>
<tr>
<td>Cube</td>
<td>0.1261</td>
</tr>
<tr>
<td>Infinite Square Prism</td>
<td>0.1399</td>
</tr>
<tr>
<td>Infinite Slab</td>
<td>0.1666</td>
</tr>
<tr>
<td>Semi-infinite Solid</td>
<td>0.1667</td>
</tr>
</tbody>
</table>

Fig. 1. Values of the coefficient \(A\) (referred to center of gravity locations) for different solid bodies.

In Figure 1 the values of \(A\) (referred to center of gravity locations) are listed for a number of body shapes that may be considered in thermal diffusivity studies. These values were calculated by using in Equation (11) the expressions of \(T\) developed by Carslaw and Jaeger (4) for these shapes. The interpretation of the characteristic dimension is also illustrated in the figure.

The coefficient \(A\) for a semi-infinite solid, the value of which was derived earlier (3), is also included in Figure 1. By comparing the last two items one may conclude that \(A = \frac{1}{4}\) holds good for any point inside a slab; or in other words that it is not affected by the other surface so long as the characteristic length is taken as the distance between the point and the nearest surface.

As pointed out in connection with Equation (1), \(\Phi\) is a correction term applicable whenever the \(\tau/t_m \to 0\) condition is not fulfilled closely enough in the experiment.

An expression derived earlier (3) for \(\Phi\) for a semi-infinite solid can be approximated as follows:

\[
\Phi_1 = 1 + \frac{1}{2} \frac{\tau}{t_m} \quad (13)
\]

which is sufficiently accurate to about \(\tau/t_m = 0.15\). In Appendix A it will be proved that Equation (13) is applicable to solids of any geometry.

The explicit form of the other correction term, function \(\Phi_2\), for semi-infinite solids can be evaluated from the graph in Figure 7 of reference 3, which was arrived at theoretically. (In this plot the product of \(ht/k\) and \(h \ell/k\) was used as the independent variable instead of the group \(h \ell/k\)). This graph indicates that for finite values of \(h \ell/k\), \(\Phi_2 > 1\). For \(h \ell/k > 10\) the following empirical equation seems to be applicable:

\[
\Phi_2 = 1 + \frac{2}{(h \ell/k)} \quad (14)
\]

The results of many computer calculations indicate that this equation is also applicable to solids of other geometries,!* at least when \(h \ell/k > 10\). In thermal diffusivity

*According to Equation (14) \(\Phi_2 \to \infty\) as \(h \ell/k \to 0\). For points at center of gravity locations in simple bodies having three planes of symmetry, this is the expected behavior of \(\Phi_2\), since \(t_m \to \infty\) as \(k \to 0\). For points in a semi-infinite solid, on the other hand, \(\Phi_2 \to 3\) as \(h \ell/k \to 0\). This may be seen from Figure 7 of reference 3.
measured. One should nevertheless endeavor to make the group \( h_0/k \) as high as possible—at least higher than 30. Methods of achieving this will be discussed.

**EXPERIMENT**

The cube is undoubtedly the most convenient shape for use as a specimen (see Figure 1). Figure 2a illustrates a cubic specimen prepared for thermal diffusivity test. To ensure high output and reduce heat conduction along the wires, very light gauze (say B and 5 gauge 30) chromel-alumel thermocouples are recommended. The twin-hole porcelain tube of \( \% \)-in. O.D. provides reliable electrical insulation for the wires, both inside and outside the specimen, and a strong handle for manipulation. Extreme care should be exercised to ensure that the thermocouple junction is at the center of the specimen and that it is in good contact with the specimen material.

Thirty seconds seems to be a convenient order of magnitude for \( t_m \). The characteristic specimen dimensions corresponding to this value have been calculated for several groups of materials and are listed in Table 1. Smaller specimens can also be used if the laboratory possesses facilities (for example, oscillograph) for recording rapid temperature changes. (It should be remembered that \( t_m \) is proportional to the square of the characteristic dimension.)

**Table 1. Characteristic Dimensions of Cubic Specimens**

<table>
<thead>
<tr>
<th>Group No.</th>
<th>Material group</th>
<th>( \ell ), cm.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Insulating materials, wood, soils</td>
<td>0.5- 1.0</td>
</tr>
<tr>
<td>2</td>
<td>Most building materials</td>
<td>0.0- 2.0</td>
</tr>
<tr>
<td>3</td>
<td>Rocks</td>
<td>1.3- 2.8</td>
</tr>
<tr>
<td>4</td>
<td>Most ceramics</td>
<td>1.2- 5.2</td>
</tr>
<tr>
<td>5</td>
<td>Iron, lead, brass</td>
<td>5.3- 9.0</td>
</tr>
<tr>
<td>6</td>
<td>Aluminum, copper, silver</td>
<td>14.0-20.0</td>
</tr>
</tbody>
</table>

Because of the large specimen sizes required, the present method is not particularly well suited for measuring the thermal diffusivity of most metals. Although fast-response recording devices permit the selection of considerably smaller specimens, with the reduction of \( \ell \) the group \( h_0/k \) also diminishes and \( t_m \) may become increasingly dependent on the function \( q_0 \) [see Equation (1)], the exact nature of which is not yet known for lower values of \( h_0/k \).

To keep the group \( h_0/k \) at a conveniently high level, it is recommended that the specimen be submerged in a liquid metal bath (primary bath) during the pretesting period (that is, for \( t < t_m \)). When the specimen acquires uniform temperature at the desired level, \( T_p \) and after the application of the temperature pulse (that is, for \( t > t_m \)), it is immaterial whether the temperature pulse is a heat pulse or a cold pulse, that is, whether \( T_p > T_0 \) or \( T_p < T_0 \). In fact, it is very convenient to start the pulse by submerging the specimen (for \( 0 < t < t_m \) in a bath of the same liquid metal, referred to as the secondary bath, at some lower temperature level, \( T_p < T_0 \)). In addition to providing high heat transfer coefficients, liquid metals also offer the advantage that they do not penetrate the pores of the specimen owing to their high contact angles.

In the DBR/NRC laboratory mercury baths are used for room temperature tests. (The experimenter should consult reference 5 concerning the toxic characteristics of liquid metals.) The secondary bath is kept at the freezing point of mercury (\(-38.5^\circ C\)) by means of an alcohol jacket cooled by occasional addition of solid carbon dioxide.

With mercury the effective value of the heat transfer coefficient in the primary bath is of the order of 0.1 cal./(sq. cm.\( \cdot \)sec.\( \cdot \)\( ^\circ \)C). This becomes several times higher when the specimen is gently shaken while submerged in the mercury. With \( h_0 \approx 0.1 \text{ cm} \cdot \text{sec.} \\text{cal.}^{-1} \text{g.}^{-1} \text{OC.}^{-1} \), the effective value of the heat transfer coefficient in the secondary bath is of the order of 0.1 cal./(sq. cm.\( \cdot \)sec.\( \cdot \)\( ^\circ \)C). Furthermore, the specimens should be submerged at intervals of 1 to 5 minutes. The time is approximately eight to ten times \( t_m \), so that if \( t_m \approx 30 \text{ sec.} \), the test can be repeated in approximately 4 to 5 min. Easy repeatability is another advantage of the present method.

Figure 3 illustrates a cubic specimen of dimensions listed in Table 1. The final specimen should be shaped to fit the primary bath (after the application of the pulse). It may be noted that with a different measurement technique (3) performed on a limestone rock specimen of cubic shape (\( \ell = 1.27 \text{ cm.} \)), the thermal diffusivity was found to be 0.0115 cal./(sq. cm.\( \cdot \)sec.\( \cdot \)\( ^\circ \)C). The density of the rock is 2.65 g./cu. cm. Its specific heat is estimated at 0.2 cal./(g.\( \cdot \)\( ^\circ \)C), so that its thermal conductivity is \( k = 0.0114 \times 2.65 \times 0.2 = 0.00006 \text{ cal.}^{-1} \text{sq. cm.}^{-1} \text{sec.}^{-1} \text{OC.}^{-1} \). Owing to the stirring of the primary bath, the heat transfer coefficient is...
probably of the order of 1.0 cal/(sq. cm/sec.°C.); thus \( h/k = 1.27/0.0060 = 212 \), which is sufficiently high to validate the assumption that \( \Phi_1 = 1.0 \).

The accuracy of this peak-time method of measuring the thermal diffusivity of solids depends on several factors: the accuracy of evaluating \( t_1 \) from the temperature-time-record; the accuracy of locating the thermocouple junction in the center of the specimen; whether a good contact has been achieved between the junction and the specimen material; the amount of foreign material (ceramic tube, thermocouple wire) along some short heat flow path; the value of the group, \( h/k, \) if it is lower than about 30.

Experience shows that \( t_1 \) can be determined to an accuracy of about 3%. Errors associated with locating the thermocouple junction and achieving good thermocouple-to-specimen contact depend entirely on the care taken in the experiment, and are difficult to assess. These errors may be reduced by radiographic inspection of the specimen. Errors associated with the fourth and fifth factors depend, to some extent, on the size of the specimen. To reduce them, the largest available piece of the material should be selected for the specimen, preferably such that \( l > 1.0 \text{ cm} \).

For specimens of elongated shape (for example, cylindrical, prismatic, ellipsoidal), it is advisable to introduce the ceramic tube and the thermocouple wires along some long heat flow path. An example is shown in Figure 2b.

It is good practice to stir the primary bath well and to check the magnitude of the Nusselt number after the calculation of thermal diffusivity.

Experience gained in the DDB/NRC laboratory indicates that for materials listed in the first group of Table 2 an overall accuracy better than 4% can be achieved if great care is exercised in preparing the specimen and in performing the test. Whenever possible, the test specimen should be machined or ground to one of the regular shapes shown in Figure 1. If, however, the material is very hard or available only in small pieces, it is possible to test irregular shaped specimens (Figure 2b). Figure 1 reveals that the range of variation of coefficient \( A \) is relatively narrow. It is possible, therefore, to estimate its value for shapes other than those listed if only an approximate value of the thermal diffusivity is of interest.

For irregular shaped specimens half of the smallest diameter of the body should be taken as the characteristic dimension \( t \). Consequently, the thermocouple junction has to be located half way between these two governing surfaces (Figure 2b) and \( A \) should be assessed on the basis of their curvature and the nearness of the other surfaces, making use of the values listed in Figure 1.

To obtain an accurate value of the thermal diffusivity of irregular shaped specimen it is necessary first to determine the value of \( A \) for the particular shape and thermocouple location from a dividing Equation (10), that is, for \( h = \infty \) by Equation (12):

\[
\Phi_1 = \frac{t_1}{t_M}
\]

At \( t_1 \) the \( \frac{dA}{dt} = 0 \); using Equation (9) one obtains

\[
\frac{\partial}{\partial t} \left( \frac{\Phi_1}{\Phi_2} \right) = \frac{\partial}{\partial t} \Phi_1 = 0
\]

The Taylor series expansion of \( \Phi_1 \) in the neighborhood of \( t_M \) is

\[
\Phi_1 = \Phi_1 + \frac{\partial \Phi_1}{\partial t} (t-t_M) + \frac{1}{2} \left( \frac{\partial^2 \Phi_1}{\partial t^2} \right) (t-t_M)^2 + \ldots
\]

where the third term on the right side of the equation is zero on account of Equation (11). By differentiating

\[
\frac{\partial}{\partial t} \left( \frac{t_1}{t_M} \right) = \frac{1}{t_M} \frac{\partial t_1}{\partial t} + \frac{1}{2} \left( \frac{\partial^2 t_1}{\partial t^2} \right) + \ldots
\]

from which

\[ t_M = \frac{1}{t_1} \]

By virtue of Equation (10) and of the fact that \( t_M = \infty \) for small values of \( t_1/t_M \) this procedure provides proof for the general applicability of Equation (13).

LITERATURE CITED


APPENDIX A: VERIFICATION OF EQUATION (13)

The defining equation for the function \( \Phi_2 \) is obtained by dividing Equation (1) with \( \Phi_1 = 1.0 \), that is, for \( h = \infty \) by Equation (12):

\[
\Phi_2 = \frac{t_1}{t_M}
\]

At \( t_1 \) the \( \frac{dA}{dt} = 0 \); using Equation (9) one obtains

\[
\frac{\partial}{\partial t} \left( \frac{\Phi_1}{\Phi_2} \right) = \frac{\partial}{\partial t} \Phi_1 = 0
\]

The Taylor series expansion of \( \Phi_1 \) in the neighborhood of \( t_M \) is

\[
\Phi_1 = \Phi_1 + \frac{\partial \Phi_1}{\partial t} (t-t_M) + \frac{1}{2} \left( \frac{\partial^2 \Phi_1}{\partial t^2} \right) (t-t_M)^2 + \ldots
\]

where the third term on the right side of the equation is zero on account of Equation (11). By differentiating

\[
\frac{\partial}{\partial t} \left( \frac{t_1}{t_M} \right) = \frac{1}{t_M} \frac{\partial t_1}{\partial t} + \frac{1}{2} \left( \frac{\partial^2 t_1}{\partial t^2} \right) + \ldots
\]

from which

\[ t_M = \frac{1}{t_1} \]

By virtue of Equation (10) and of the fact that \( t_M = \infty \) for small values of \( t_1/t_M \) this procedure provides proof for the general applicability of Equation (13).
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