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AN APPARATUS FOR MEASURING THERMAL DIFFUSIVITY IN AIR

V.V. Mirkovich

DECEMBER 1976

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
CANMET REPORT 77-21

Available by mail from:

Printing and Publishing
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Ottawa, Canada K1A 0S9

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Catalogue No. M38–13/77-21 Price: Canada: $1.25
ISBN 0-662-00746-8 Other countries: $1.50

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Nº de catalogue M38–13/77-21 Prix: Canada: $1.25
ISBN 0-662-00746-8 Autres pays: $1.50

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AN APPARATUS FOR MEASURING THERMAL DIFFUSIVITY IN AIR

by

V.V. Mirkovich*

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ABSTRACT

In view of the recent interest in materials for generating and conserving energy, the determination of their thermal properties has become important. One such property is thermal diffusivity. Two prototypes for measuring thermal diffusivity of solid materials in air are described. These prototypes, which are based on the concepts of an infinite cylinder and radial heat flow, are designed to measure thermal diffusivity in transitory and periodic temperature modes. Measurements performed on Pyroceram Code 9606 have shown that accurate data can be obtained in one of the experimental models in the temperature range of 25° to 700°C.

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UN APPAREIL POUR MESURER LA DIFFUSIBILITE THERMIQUE DANS L'AIR

par

V.V. Mirkovich*

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SOMMAIRE

La crise d'énergie, qui sévit actuellement, a suscité un intérêt grandissant envers les matériaux qui produisent et conservent l'énergie; par conséquent il est important de connaître leurs propriétés thermiques. L'une d'entre elles est la diffusibilité thermique. Dans le présent rapport, l'auteur décrit deux prototypes qui servent à mesurer la diffusibilité thermique de matériaux solides dans l'air. Ces deux prototypes, dont la conception s'inspire de cylindres infinis et d'un écoulement de chaleur radial, sont ainsi faits dans le but de calculer la diffusibilité thermique selon des méthodes de température périodiques et transitoires. Les mesures recueillies à l'aide du Pyroceram Code 9606 démontrèrent qu'un des modèles d'essais peut fournir des données précises dans l'échelle de température variant entre 25^{o} et 700^{o}C.

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INTRODUCTION

The recent critical energy shortage encountered by the high energy consuming nations of the world has underlined the need for new and improved methods of generating and conserving energy. This need has led to an increased interest in the thermal properties of materials. Specifically, with respect to the materials under investigation in the Industrial Minerals Laboratory, the need for thermal diffusivity data has been recognized. Therefore, a project was undertaken to develop a thermal diffusivity measuring apparatus capable of meeting the particular requirements of these materials.

Various thermal diffusivity devices are described in the literature. The variety of these devices should not be attributed only to the experimentalist's desire to introduce novelty or to demonstrate ingenuity by devising physical methods that satisfy one of the numerous theoretical boundary conditions of the non-steady-state heat flow. Rather, it should be attributed to the wide range of physical properties of materials. To obtain the highest precision, accuracy and speed of measurement, the method and the design of the measuring devices often have to be chosen to satisfy as best as possible a particular material or a range of materials.

In general, experimental methods for measuring thermal diffusivity can be divided into two categories: periodic temperature methods and transitory temperature methods. Furthermore, depending on the shape of the specimen, each of these broad

categories can be further subdivided. Among the first and probably the best known of the periodic methods, was one developed by Ångstrom[1] in which the principle of a semi-infinite rod was employed. This popular method was modified and improved by various authors[2,3,4,5,6] to suit different conditions of measurement and/or specific requirements imposed by their materials. Other periodic methods based on the concept of a thin plate[7,8], a semi-infinite solid[9], and a cylinder[10] were developed for materials where the form of a thin, long rod was not suitable. Similarly, various transitory temperature methods were developed. These are also characterized by the geometrical configuration of the specimen, such as long rods[11], flat plates[12,13] and cylinders[14,15].

The requirements imposed by the materials to be investigated in this laboratory are such that none of the above devices would be entirely satisfactory. It should be obvious that in the case of coarse-grained materials, such as some rocks and minerals, small samples in the form of thin plates, as used in the very versatile flash method[13], would be too small to integrate the contribution of individual grains toward the overall thermal diffusivity of the specimen. On the other hand, an apparatus using layer specimens but based on uni-directional heat flow, as in the case of the semi-infinite solid, would be of limited value for low thermal diffusivity materials. The accuracy of results in this case would be low because of the rapid attenuation of the heat wave. For instance, in a semi-infinite body with thermal diffusivity of 0.01 $cm^2/sec.$, the attenuation of a linear flow

heat wave with a 60-second cycle is 90 per cent at the distance of 1 cm from the surface.

Minimum heat wave attenuation is attainable in a spherical specimen with radial heat flow toward the centre of the sphere. Experimentally, however, that is impractical because of the difficulty of sample preparation and also because of the even greater difficulty of generating heat pulses of equal magnitude over the whole surface of the sphere. It was decided therefore that the design of the thermal diffusivity apparatus should be based on the concepts of an infinite cylinder and radial heat flow. Furthermore, it should be constructed in such a way as to allow measurements to be performed in both transitory and periodic temperature modes. The ability to measure thermal diffusivity in two temperature modes, plus the fact that a cylindrical model under conditions of radial heat flow is characterized by virtual freedom from spurious heat losses, would not only increase the confidence in the accuracy of the results, but also extend the range of thermal diffusivities in which the measurements could be performed.

THEORETICAL CONCEPTS

The mathematical boundary conditions of the theoretical thermal diffusivity models have previously been discussed in detail[16]. Briefly, two models of heat flow are being considered in the experimental measurements. In both cases the material is considered to be isotropic and its thermal properties are indepen-

dent of temperature over a limited temperature span.

For materials subject to oxidation, it is advantageous that thermal diffusivity be measured in vacuum. However, it is very difficult to obtain accurate readings in vacuum unless the thermocouples are in perfect contact with the material. Even so, it was demonstrated by Maglic[17] that intrinsic thermocouples, i.e., thermocouples that are welded to a conductive specimen that then becomes part of the measuring circuit, can have functions of different heat capacities and consequently exhibit different response times for the same temperature pulse. Thus for the measurement of thermal diffusivity of non-metallic and/or electrically non-conductive materials, where perfect thermal contact between the thermocouple and the material cannot be obtained, air or some inert gas could be used as a transfer medium. In this case the thermocouples are placed in their respective thermocouple holes without any particular effort to obtain good contact with the material, and the heat pulses are transferred from the specimen to the thermocouple through the air present around the hot-junction of the thermocouple. Furthermore, some natural rocks, minerals and cementitious materials contain water and volatiles, the release of which would first, alter the nature of the material, and second, make it impossible to maintain the necessary vacuum.

It was decided therefore to design and construct an apparatus capable of measuring thermal diffusivity of solids in air.

In the transitory temperature measuring method, herein designated Model 1, the temperature is measured at the centre and

at a point, r, near the surface of the infinite cylinder of radius a. The initial temperature of the cylinder is zero and its surface temperature is a linear function of time, t. By subtracting the equation representing the temperature at the centre of the cylinder from the equation representing the temperature at the point near the surface of the cylinder, one arrives at an expression that relates the change of dimensionless temperature differential between the two measuring points, V, to a factor, $\kappa t/a^2$, where κ is thermal diffusivity. With constantly increasing surface temperature, as $\kappa t/a^2$ increases from 0 to infinity, V increases asymptotically from 0 to 1. The position of the curve for a given V and $\kappa t/a^2$ is also dependent on the ratio r/a. To obtain thermal diffusivity from an experimental plot, such as shown in Figure 1, the time necessary to reach some convenient value of the relative temperature, say V = 0.5, the "half time", must be determined. Once t is known, thermal diffusivity can be calculated from the following expression which gives $\kappa t/a^2$ as a function of r/a for $1 \geq r/a \geq 0.5$ for V = 0.5:

$$\kappa t/a^2 = 0.198 + 0.00747(r/a) - 0.0687(r/a)^2$$

In measuring thermal diffusivity in a periodic temperature mode, Model 2 method, the temperature is measured at the centre and near the surface of the infinite cylinder while its surface is exposed to a sinusoidal temperature wave. The time of one full cycle of the temperature wave is t_c. The time lag for a given temperature wave to advance from the surface measuring point to the centre is Δt_c. By equating expressions representing the temperatures at the point near the surface and at the centre,

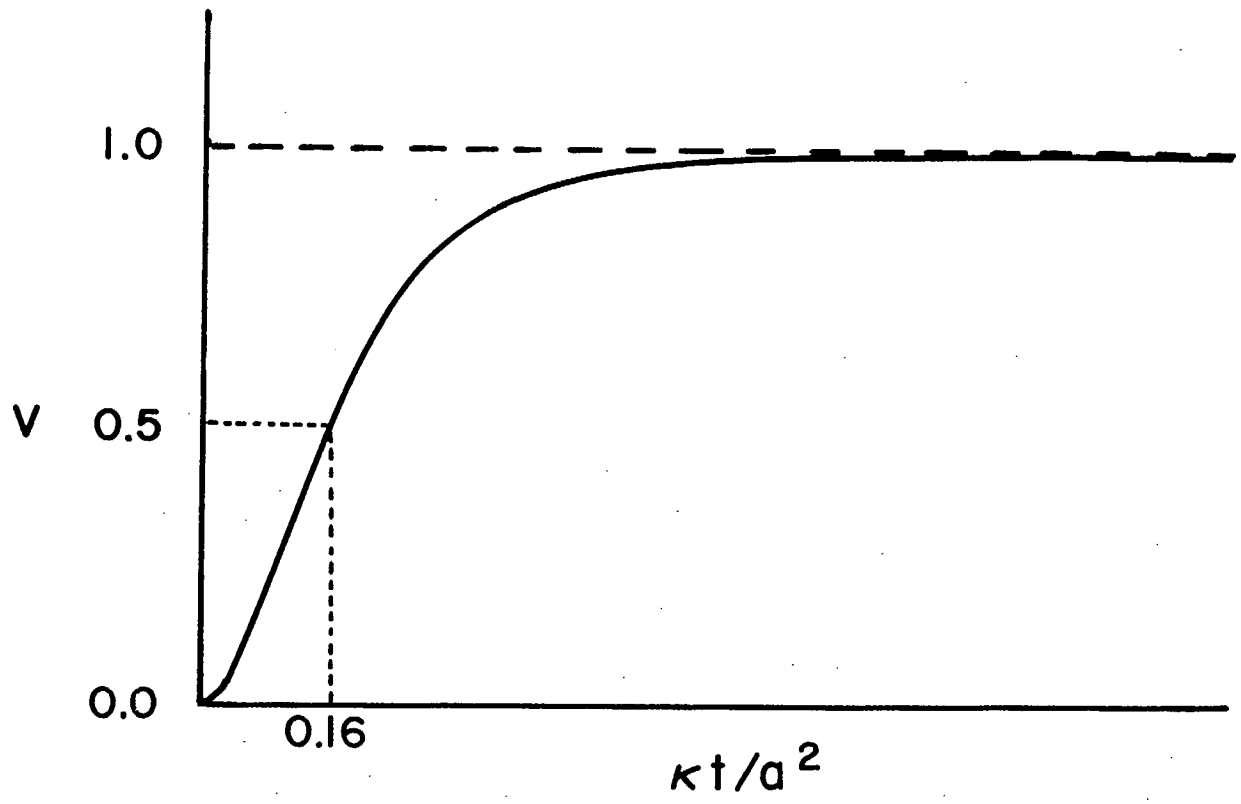

Figure 1. Plot of relative temperature, V, versus dimensionless factor, $\kappa t/a^2$.

one obtains $\omega(\Delta t_c) = \theta_o(\omega' a)$ where $\omega = 2\pi/t_c$, $\omega' = (1/\kappa)^{0.5}(2\pi/t_c)^{0.5}$, and θ_o is a function related to Bessel functions. Radius, a, in the above equation is now the distance from the centre to the outside measuring point. Thermal diffusivity is obtained by determining the time lag, Δt_c, from the experimental plot in which the sinusoidal temperatures of the centre and the side measuring points were simultaneously recorded. From $\omega(\Delta t_c)$ one calculates $(\omega' a)$, from which in turn thermal diffusivity can be obtained.

REFERENCE MATERIAL

To determine the accuracy of measurement of a new apparatus, a well-characterized standard is a most important prerequisite. In the field of thermal conductivity and thermal diffusivity, universally recognized standards are still not available. However, for various reasons some materials have been used by different researchers and their characteristics and values have been reported. Thermal diffusivity of Pyroceram Code 9606* was measured by several investigators[12,18,19] and their results are in good agreement with each other. The author also measured the thermal diffusivity of Pyroceram[20]. The measurements were performed in vacuum in transitory and periodic modes. The results were in reasonable agreement with the work referred to above.

Because the range of thermal diffusivities of materials to be investigated is expected to be of the same order of magnitude as that of the Pyroceram, and because of good characterization, resistance to oxidation at higher temperatures, and availability of suitable samples, Pyroceram was selected as the reference material for this study.

THE APPARATUS

Two experimental thermal diffusivity measuring devices were constructed, each designed to operate in both the transitory

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* Pyroceram Code 9606 is a polycrystalline material manufactured by Corning Glass Company, Corning, N.Y., U.S.A.

and periodic modes. The measuring circuitry and power supply system are the same for both.

a) Measuring Circuitry and Power Supply

Temperatures in the specimens are measured with two chromel-alumel thermocouples (28 B&S gauge). The temperature measuring system is designed so that either absolute output of each thermocouple or the emf difference between the two thermocouples can be recorded. The arrangement is schematically shown in Figure 2.

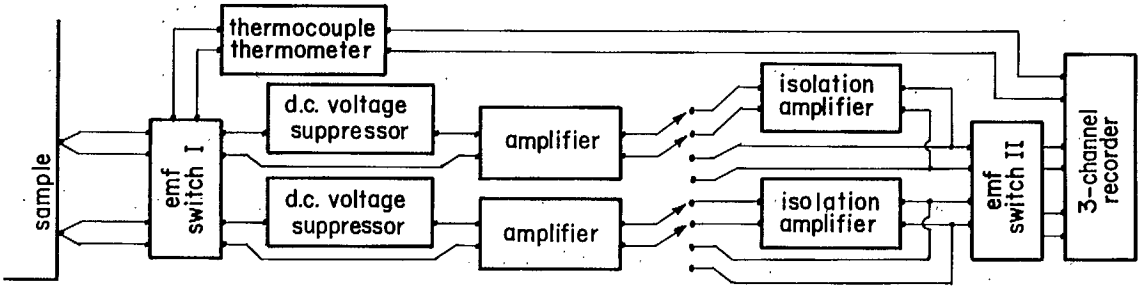

Figure 2. Block diagram of the measuring equipment.

In either case, the output of the thermocouples is fed into the emf switch I, from which the signal of either of the thermocouples can be directed, first to a Digitec Model 590 KC, Type K* digital thermocouple thermometer for direct reading of the sample temperature, and then further routed to channel 3 of the RKD Model B-34** potentiometric type 3-pen recorder. Both

---

* United Systems Corporation, Dayton, Ohio, U.S.A.
**RDK Rikadenki Kogyo Co.; 9-11 Chome Chochu Meguroku, Tokyo, Japan.

the thermocouple thermometer and the 3-channel recorder are null-balancing instruments not affecting the thermocouples' emf outputs.

To record in the absolute mode (i.e., to perform the measurements in Model 2), the non-periodic portion of the total output of each thermocouple from the emf switch is cancelled by means of a Model DAV-46D* dc voltage suppressor installed in each thermocouple circuit. The periodic overflow of each thermocouple is fed into a RKD Model A-10** low-noise dc amplifier, then to a H-P Model 8875A*** isolation amplifier, then to the emf switch II, and finally to the 3-pen recorder. Two channels are used for recording the amplified periodic portions of the thermocouple outputs, while the third channel registers the direct output from one of the thermocouples.

For measurements in the differential mode, voltage suppressors are used once again to cancel the basic output of the thermocouples while the transitory portion is fed to the amplifiers. By means of the emf switch II, the positive legs of the two circuits are joined while the negative legs are connected to one of the recording channels.

Differential temperature measurements can be made with differential thermocouples if the hot joints are electrically isolated from each other. However, some non-conductors are known to become electrically conductive at higher temperatures, or measurements may be necessary on an electrically conductive material.

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* General Resistance Inc.; Mt. Vernon, N.Y., U.S.A.
** RDK Rikadenki Kogyo Co.; 9-11 Chome Chochu Meguroku, Tokyo, Japan.
***Hewlett-Packard, 195 Page Mill Rd.; Palo Alto. California, U.S.A.

In such cases, measurements cannot be made because the differential emf is short-circuited through the conductor. As the negative side of the low-noise amplifiers in this arrangement is grounded, the input to the amplifier is not isolated from its output, and consequently, direct differential measurement on electrically conductive materials is not possible. To overcome this difficulty, one-to-one isolation amplifiers are installed between the low-noise amplifiers and the emf switch II. When not needed, the isolation amplifiers can be excluded from the measuring circuitry.

Two heat sources are used in the experimental devices. To maintain the temperature of the specimen at a desired level, power from a variable transformer is fed at a constant rate to one of the Nichrome heater wires. The heat pulses are obtained by using a Wavetek Model 114* voltage function generator. The output from this generator is fed to a direct current power amplifier, whose output in turn is supplied to the other Nichrome heater wire.

b) Apparatus I: Heaters Separate from Specimen

The Apparatus I is schematically shown in Figure 3. The cylindrical sample (I), 25.4 mm in diameter and 70 to 80 mm high, composed of several discs of the same material stacked on top of each other, is held between the upper (D) and lower (J) cylindrical insulators, and is centred within the fused-silica

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* Wavetek, 9045 Balboa Ave.; San Diego, California, U.S.A.

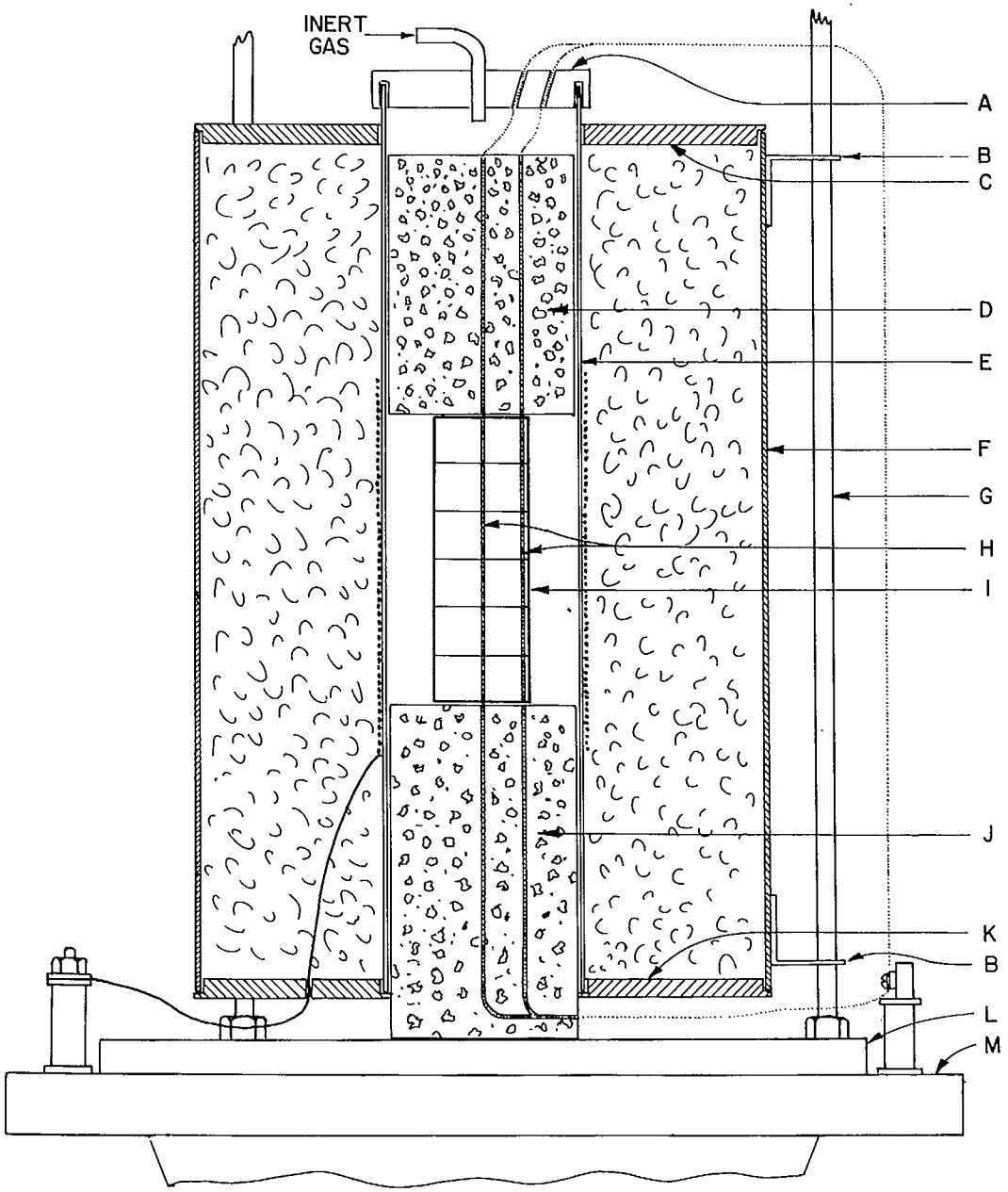

Figure 3. Schematic diagram of Apparatus I for measuring thermal diffusivity of solids in air. Heaters are remote from the specimen. A = ceramic cover, B = guiding bracket, C = upper Transite cover plate, D = upper cylindrical insulator, E = fused-silica tube, F = stainless steel furnace casing, G = steel guide rods, H = thermocouple hole, I = cylindrical sample, J = lower cylindrical insulator, K = lower Transite cover plate, L = main Transite plate, M = metal flange.

tube (E). The fused-silica tube, 43.5-mm OD, 40.5-mm ID and 315 mm long, with two lengths of non-inductively wound Nichrome wire on the outside, forms both the heater and heat pulse generator. Two 5-metre long, 24 B&S (0.51-mm diameter) Nichrome wires are wound into four congruent grooves, cut on the outside of the tube in the form of a helix, over 12 cm of the tube. The pitch of any one of the four grooves is four turns per 25-mm length of the tube. To maintain good electrical separation between the Nichrome wires, a thin layer of refractory cement is applied to the section of the fused-silica tube over which the wires are wound. The fused-silica tube is held in position by means of the upper (C) and lower (K) Transite cover plates, which are in turn affixed to the 140-mm diameter, 295-mm high stainless steel furnace casing (F). The furnace assembly can be raised and lowered on three steel rods (G) by means of guiding brackets (B) attached to the stainless-steel casing. The three steel rods are bolted to a supporting metal flange (M), holding at the same time the main Transite plate (L) in position over the metal flange. The metal flange is part of a vacuum system described elsewhere[16] and is usually not used in this arrangement. The leads from the two Nichrome-wire heaters are brought out of the furnace through the lower Transite cover plate and are attached to the power supply terminals on the supporting metal flange. The thermocouples are butt-welded and the weld beads are jammed into the 0.75-mm diameter thermocouple holes of the central disc specimen. One pair of thermocouple legs of equal polarity is taken out of the furnace through the bottom cylindrical insulator and the other pair is

led out through the upper insulator and the ceramic cover (A). The thermocouples are attached to the terminal post located on the supporting metal flange. The temperature is measured at the centre of the cylinder and at a radial distance of 11.5 mm.

To provide the required thermal insulation, the annular space between the fused-silica tube and the stainless steel casing is filled with granular expanded vermiculite.

Should it become necessary, the apparatus can be readily adapted to measure in inert atmospheres. In such a case, after the sample column has been assembled inside the furnace, the three steel guide rods are removed and the apparatus is covered with a glass jar that fits onto the metal flange of the vacuum system.

After the air is evacuated from the apparatus (the main Transite plate (L) is perforated), inert gas is allowed to fill the system. The glass jar can then be removed and the inert gas slowly fed through the opening in the ceramic cover on top of the fused-silica tube.

The principal advantage of this design is the relative ease of arranging the samples and setting up the sample column in the furnace. This feature is important when considering that the time necessary to make a thermal diffusivity measurement is usually short (a matter of a few minutes, at most) in comparison with the time required to assemble the specimens within the apparatus. Unfortunately, after extensive experimentation, it became apparent that this measuring system was not functioning as expected. It appeared that the transient power output, i.e., the

power output from the heat-pulse generating coil was insufficient. To overcome this difficulty, an apparatus was designed in which the pulse-generating coil was wound directly onto the surface of the specimen.

c) Apparatus II: One Heater Wound Directly onto the Specimen

The schematic diagram of Apparatus II is shown in Figure 4. The sample column, 25.4 mm in diameter and some 70 to 80 mm long, is composed, as before, of several discs (G) of the same material. To prevent axial losses, a cylindrical ceramic insulator (D) is placed on each end. The column is held together by two pairs of stainless steel wires (not shown) binding the cylindrical insulators. A 0.51-mm Nichrome heating wire (F) is non-inductively wound in a helix around the sample column and partially over the cylindrical insulators. The wire is covered with a thin layer of refractory cement. The principal reason for applying the refractory cement is to keep the wires separate from each other and thus prevent electrical short-circuiting. Two thermocouple holes (H) extend through one insulator, two sample discs and halfway into the third disc.

To maintain the desired temperature level at which measurements are to be performed, the specimen assembly is placed into a 41-mm ID, 500-mm long mullite tube (A). This horizontally placed tube with a non-inductively wound Nichrome heater (E) on its outside, covered with some 30 mm of ceramic insulation (C), serves as an electric furnace. With thermocouples (I) inserted into their respective thermocouple holes, the specimen assembly is

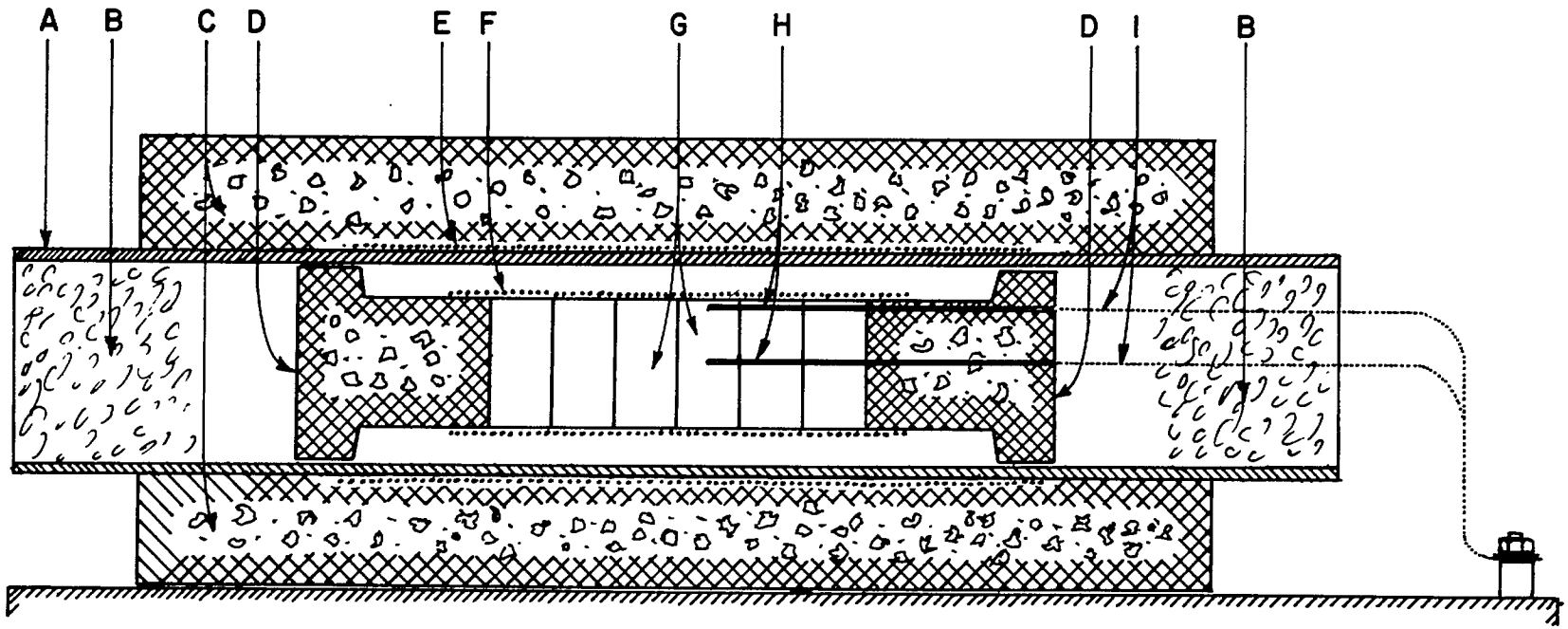

Figure 4. Schematic diagram of Apparatus II for measuring thermal diffusivity of solids in air. Heat-pulse generating wire heater wound directly onto the specimen. A = mullite tube, B = fibrous ceramic insulation, C = ceramic insulation, D = cylindrical ceramic insulator, E = Nichrome heater, F = Nichrome heating wire, G = sample disc, H = Thermocouple hole, I = thermocouple.

positioned inside the mullite tube. After completing the electrical connections, the ends of the mullite tube are closed with a fibrous ceramic insulation (B).

RESULTS AND DISCUSSION

The results obtained in measurements with Apparatus I of the thermal diffusivity of Pyroceram 9606 in the temperature range of 25°C to 900°C revealed a substantial deficiency in its design. The data obtained in the Model I measuring method (transitory heat flow) were about one order of magnitude lower than the data reported in the literature[12,18,19] and were generally scattered. The accuracy of the Model II measuring method (periodic heat flow) was considerably better. On the average, however, the results were some 13 per cent higher than the values given in the above references.

After numerous measurements at different temperature levels, in which the effects of all variables were individually examined, it was concluded that the principal factor responsible for the inaccuracy of the results was the relatively high heat capacity of the heat-pulse generating system. For example, whereas the heat-pulse response to an electric signal of a 0.17-mm thick tungsten wire freely suspended in vacuum will occur in a matter of milliseconds, the response of a 0.51-mm thick Nichrome wire placed onto the surface of a fused-silica tube and covered with refractory ceramic cement is more likely to take 5 or 10 seconds more. Consequently, the tungsten wire will almost instantaneously

reach the maximum temperature and the heat radiated from it onto the surface of the specimen will be constant. (Admittedly, the temperature difference between the tungsten wire and the specimen's surface decreases as the temperature of the specimen increases, but the increase of the specimen temperature by some 10° to 20°C is experimentally insignificant in comparison with the overall sample-to-tungsten wire temperature difference of 1000° to 2000°C).

Such a case is schematically presented in Figure 5, where the surface temperature of an infinite cylinder heated by a constant-temperature heat source is shown to rise at a constant rate. Figure 6 shows how, for this case, the temperature difference between the surface and the centre of the cylinder increases before reaching the maximum. On the other hand, for a heat-pulse generator made of Nichrome wire wound on a fused-silica tube, because of the relatively large heat capacity, the maximum temperature of the heater assembly is reached only after an extended period of time. Because of this, the rate of cylinder surface temperature increase also increases until the heat-pulse generator reaches its maximum temperature (Figure 7). The centre temperature increases at a lower rate, and continues to do so until after the rate of the surface temperature increase becomes constant. The resulting temperature difference is shown in Figure 8. As its rate of increase is substantially smaller than that shown in Figure 6, the "half time" for the curve to reach the maximum is considerably longer, which, in turn gives erroneously low thermal diffusivity values.

Regarding the results obtained with the Model 2 measuring

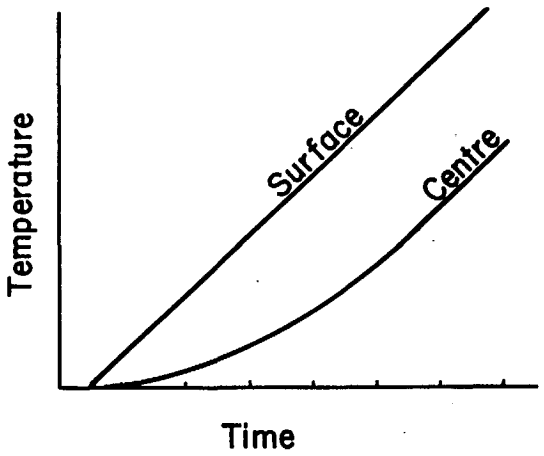

Figure 5. Surface and centre temperature changes of an infinite cylinder uniformly exposed to a constant temperature heat source.

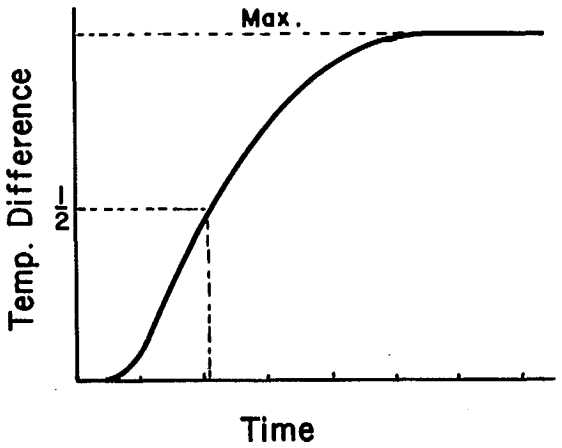

Figure 6. Temperature difference increase between the surface and centre of an infinite cylinder uniformly exposed to a constant temperature heat source.

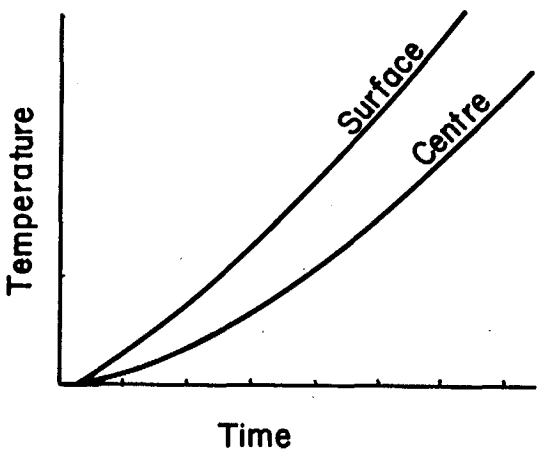

Figure 7. Surface and centre temperature changes of an infinite cylinder uniformly exposed to a rising temperature heat source.

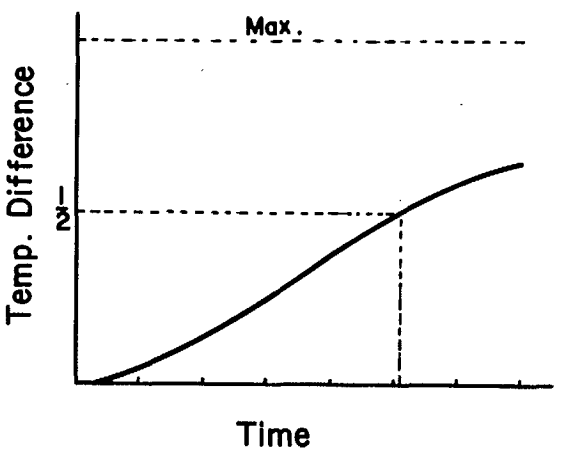

Figure 8. Temperature difference increase between the surface and centre of an infinite cylinder uniformly exposed to a rising temperature heat source.

method, it is obvious that the relatively high heat capacity of the heat-pulse generating system was responsible for decreasing the measured time lag of the temperature wave (Δt_c), thus producing data 13 per cent higher than the reported literature values. Unfortunately, there is no plausible explanation for this phenomenon at the present time.

The results obtained with Apparatus II show a substantial improvement over those measured with Apparatus I. The measurements performed with the Model 2 measuring method in the temperature range of 200° to 700°C are in very good agreement with published data. The results obtained in Model 1 for the temperature range of 25° to 700°C are, however, somewhat higher than the literature values. This seems to be characteristic for measurements in Model 1 with the heat-pulse generator wound directly onto the surface of the cylinder, because all results are displaced by a constant amount. A possible cause may be electrical because, with heater windings on the surface of the sample, it is not possible to shield the thermocouples from stray emf's. Another cause could be the effect of the specimen on the temperature of the heat-pulse generator. By adjusting the $\kappa t/a^2$ factor (from $\kappa t/a^2 = 0.147$, as calculated for the dimensions of the present samples, to $\kappa t/a^2 = 0.097$) so that the low temperature data of Model 1 coincide with the published low temperature data, an excellent agreement for all temperatures to 700°C is obtained with Rudkin's results[18]. The best agreement between the three published values is at room temperature. This can be seen in Figure 9 in which the curves calculated from Model 1 and Model 2

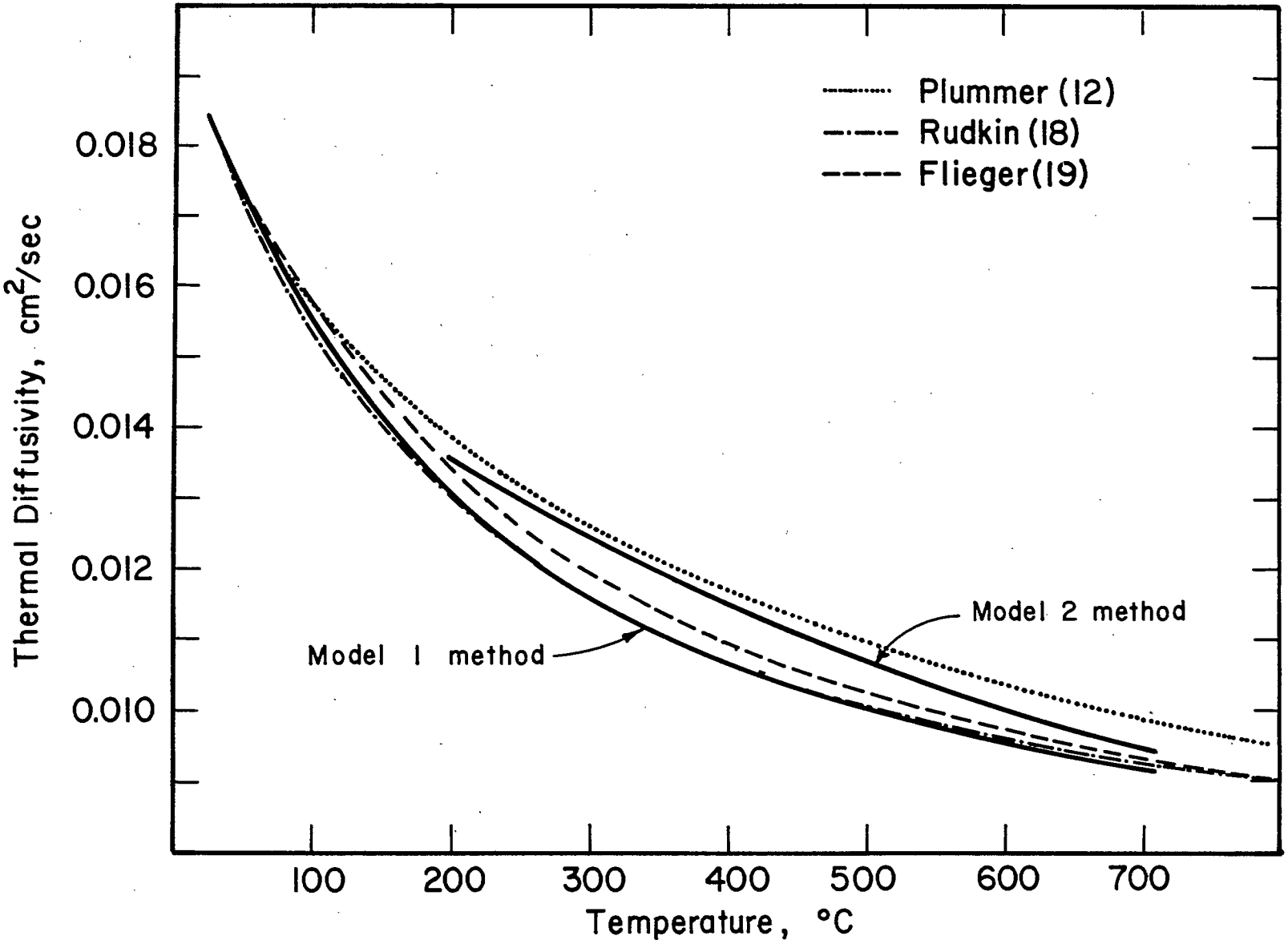

Figure 9. Thermal Diffusivity of Pyroceram Code 9606.

thermal diffusivity data obtained with Apparatus II are superimposed over the curves representing published results.

Model 1 and Model 2 data obtained with Apparatus II are tabulated in Table 1 and Table 2 respectively.

Table 3 gives the smoothed values at 25°C intervals for Model 1 data calculated from the following equation:

$$\kappa = 2.01 \times 10^{-2} - 5.68 \times 10^{-5}\theta + 1.28 \times 10^{-7}\theta^2 - 1.35 \times 10^{-10}\theta^3 + 5.26 \times 10^{-14}\theta^4$$

Table 4 gives the smoothed values at 25°C intervals for Model 2 data calculated from the following equation:

$$\kappa = 1.61 \times 10^{-2} - 1.37 \times 10^{-5}\theta + 5.85 \times 10^{-9}\theta^2$$

where (in both equations) κ is thermal diffusivity in cm^2/sec and and θ is temperature in degrees Celsius. The standard deviation for Model 1 data is ±1.4%.

For Model 2 data the standard deviation is ±1.8%. In either case the precision of the measurements is high. The accuracy of results is estimated to be ±5%.

The temperature of 700°C was not arbitrarily selected as the top limit at which measurements were to be made for this study. This limit was imposed by the nature of the heat-pulse generator used in Apparatus II. The basic requirement of the Model I measuring system is that the rate of the cylinder temperature increase is constant. In Apparatus II, this is effectively accomplished at lower temperatures by making the temperature of the heat-pulse generator constant but at an appreciably higher level than that of the cylindrical surface. However, as the surface temperature of the specimen approaches that of the heat-

TABLE 1

Thermal Diffusivity of Pyroceram Code 9606

Measured in Model 1 Measuring Method.

t° C	κ[cm^2/sec]	t° C	κ[cm^2/sec]	t° C	κ[cm^2/sec]
27	0.0187	112	0.0150	418	0.0105
40	0.0180	116	0.0154	430	0.0105
47	0.0171	125	0.0147	455	0.0100
50	0.0175	135	0.0147	455	0.0102
62	0.0173	134	0.0144	500	0.0100
64	0.0175	163	0.0138	510	0.0103
72	0.0167	172	0.0135	520	0.0103
82	0.0161	191	0.0131	530	0.0099
82	0.0162	200	0.0127	593	0.0098
84	0.0165	200	0.0131	595	0.0096
90	0.0158	248	0.0117	694	0.0092
99	0.0154	256	0.0115	695	0.0094
104	0.0157	258	0.0120	702	0.0090

TABLE 2

Thermal Diffusivity of Pyroceram Code 9606

Measured in Model 2 Measuring Method

t°C	κ[cm^2/sec]	t° C	κ [cm^2/sec]	t° C	κ[cm^2/sec]
205	0.0136	348	0.0112	512	0.0111
256	0.0127	403	0.0113	593	0.0101
283	0.0131	404	0.0114	593	0.0101
283	0.0129	404	0.0113	652	0.0093
344	0.0120	408	0.0119	684	0.0096
345	0.0123	431	0.0109		
345	0.0124	451	0.0114		

TABLE 3

Smoothed Values of Thermal Diffusivities of Pyroceram Code 9606 for Measurements in Model 1 Measuring Method.

t°C	κ[cm^2/sec]	t°C	κ[cm^2/sec]	t°C	κ[cm^2/sec]
25	0.0188	275	0.0117	525	0.0100
50	0.0176	300	0.0114	550	0.0099
75	0.0165	325	0.0111	575	0.0098
100	0.0156	350	0.0109	600	0.0097
125	0.0148	375	0.0107	625	0.0096
150	0.0141	400	0.0105	650	0.0095
175	0.0134	425	0.0104	675	0.0094
200	0.0129	450	0.0103	700	0.0092
225	0.0124	475	0.0102		
250	0.0120	500	0.0101		

TABLE 4

Smoothed Values of Thermal Diffusivities of Pyroceram Code 9606 for Measurements in Model 2 Measuring Method.

t°C	κ[cm^2/sec]	t°C	κ[cm^2/sec]	t°C	κ[cm^2/sec]
200	0.0136	375	0.0118	550	0.0103
225	0.0133	400	0.0116	575	0.0102
250	0.0130	425	0.0113	600	0.0100
275	0.0128	450	0.0111	625	0.0098
300	0.0125	475	0.0109	650	0.0097
325	0.0123	500	0.0107	675	0.0095
350	0.0120	525	0.0105	700	0.0094

pulse generator, the rate of heat transfer to the specimen progressively decreases and the rate of cylinder temperature increase is proportionately reduced. Thus, during measurements above 700°C, the rate of temperature increase of the specimen surface is no longer effectively constant, but decreases significantly over the time interval required for measurement. This condition, which is opposite to the one presented in Figure 6, is illustrated in Figure 10. Here, the rate of surface temperature increase is initially constant but, as the measurement progresses, it starts to decline. The rate of increase of the temperature of the centre initially increases, then becomes constant, and, if the measurement is carried for a sufficiently long time, may even decline. The resulting temperature difference is shown in Figure 11. It initially increases at what appears to be a normal rate, reaches a maximum and then, instead of remaining constant, it begins to decline. (The dotted line indicates how the temperature difference would have increased had the heating rate of the surface been constant). The results calculated from such measurements are erroneous and too high.

It should be noted that measurements above 700°C in Model 2 with Apparatus II become unreliable as well. As the heat-pulse generator begins to approach its maximum operating temperature a distortion in the heat wave occurs, which makes evaluation of the wave time-lag (Δt_c) difficult and unreliable.

Finally, it can be stated that measurements of thermal diffusivity in Model 1 and Model 2 methods with Apparatus II will produce accurate data in the temperature range from 25° to 700°C

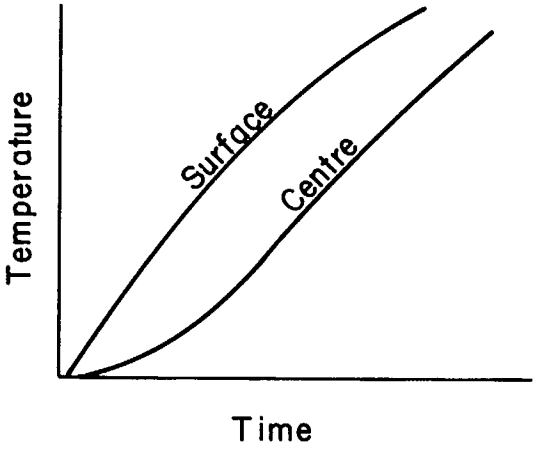

Figure 10. Temperature changes in an infinite cylinder with declining rate of surface temperature increase.

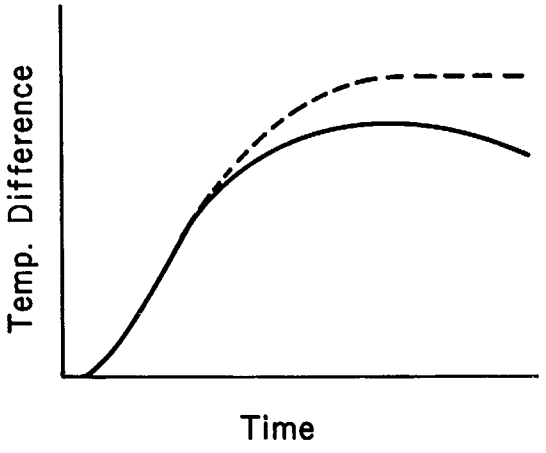

Figure 11. Change of temperature difference between the surface and the centre of an infinite cylinder with declining rate of surface temperature increase. Dotted line presents a condition of constant rate of surface temperature increase.

on materials with thermal diffusivities similar to or lower than that of Pyroceram 9606. For materials with substantially higher thermal diffusivity, the effect of the heat capacity of the heat-pulse generating system will probably be more pronounced. To establish its limitations, the system should be tested on such material. Furthermore, for purposes of scaling-up the apparatus so that measurements could be performed on coarse-grained materials such as concrete, and to avoid the cumbersome preparation of heat-pulse generator-cum-specimen, a low heat capacity heat-pulse generator for Apparatus I, capable of operating in air or inert atmosphere, should be developed.

CONCLUSIONS

The following conclusions were drawn on the basis of experimental evidence of this study:

1. Accurate results can be obtained with Apparatus II by either transitory or periodic measuring methods carried out in air.
2. Because of the nature of the heat-pulse generator in Apparatus II, the measurements at temperatures higher than 700°C are not reliable. Also, an empirical correction factor must be used with results obtained in the transitory method measurements.
3. The relatively large heat capacity of the heat-pulse generator in Apparatus I is the principal weakness of this design. A low heat capacity heat-pulse generator should be developed. Such a generator would permit scaling-up of Apparatus I.

ACKNOWLEDGEMENT

The author wishes to express his appreciation to A.G. McDonald for his generous help with the experimental part of this work and for numerous constructive discussions which helped to clarify many ambiguous points.

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