Designation: E 1461 – 01

Standard Test Method for
Thermal Diffusivity by the Flash Method[1]

This standard is issued under the fixed designation E 1461; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the thermal diffusivity of primarily homogeneous isotropic solid materials. Thermal diffusivity values ranging from 10^{-7} to 10^{-3} m^2/s are readily measurable by this test method from about 75 to 2800 K.

1.2 This test method is a more detailed form of Test Method C 714, but has applicability to much wider ranges of materials, applications, and temperatures, with improved accuracy of measurements.

1.3 This test method is applicable to the measurements performed on materials opaque to the spectrum of the energy pulse, but with special precautions can be used on fully or partially transparent materials (see Appendix X1).

1.4 This test method is intended to allow a wide variety of apparatus designs. It is not practical in a test method of this type to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without pertinent technical knowledge, or to stop or restrict research and development for improvements in the basic technique.

1.5 This test method is applicable to the measurements performed on essentially fully dense materials; however, in some cases it has shown to produce acceptable results when used with porous samples. Since the magnitude of porosity, pore shapes, sizes and parameters of pore distribution influence the behavior of the thermal diffusivity, extreme caution must be exercised when analyzing data. Special caution is advised when other properties, such as thermal conductivity, are derived from thermal diffusivity obtained by this method.

1.6 This test method can be considered an absolute (or primary) method of measurement, since no reference standards are required. It is advisable to use reference materials to verify the performance of the instrument used.

1.7 This method is applicable only for homogeneous solid materials, in the strictest sense; however, in some cases it has shown to produce data which may be useful in certain applications.

1.7.1 *Testing of Composite Materials*—When substantial inhomogeneity and anisotropy is present in a material, the thermal diffusivity data obtained with this method may be substantially in error. Nevertheless, such data, while usually lacking absolute accuracy, may be useful in comparing materials of similar structure. Extreme caution must be exercised when related properties, such as thermal conductivity, are derived, as composites may have heat flow patterns substantially different than uniaxial.

1.7.2 *Testing Liquids*—This method has found an especially useful application in determining thermal diffusivity of molten materials. For this technique, specially constructed sample enclosures must be used.

1.7.3 *Testing Layered Materials*—This method has also been extended to test certain layered structures made of dissimilar materials, where one of the layers is considered unknown. In some cases, contact conductance of the interface may also be determined.

1.8 The values stated in SI units are to be regarded as the standard.

1.9 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 714 Test Method for Thermal Diffusivity of Carbon and Graphite by a Thermal Pulse Method[2]

E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *thermal conductivity, λ, of a solid material*—the time rate of steady heat flow through unit thickness of an infinite slab of a homogeneous material in a direction perpendicular to the surface, induced by unit temperature difference. The property must be identified with a specific mean temperature, since it varies with temperature.

3.1.2 *thermal diffusivity, α, of a solid material*—the property given by the thermal conductivity divided by the product of the density and heat capacity per unit mass.

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[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.05 on Thermophysical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

3.2 *Description of Symbols and Units Specific to This Standard:*

3.2.1 D—diameter, meters.

3.2.2 k—constant in solution to diffusion equation.

3.2.3 L—specimen thickness, meters.

3.2.4 t—response time, seconds.

3.2.5 t^*—dimensionless time ($t^* = 4\alpha_s\, t/D_T^2$).

3.2.6 T—temperature, Kelvin.

3.2.7 α—thermal diffusivity, m^2/s.

3.2.8 λ—thermal conductivity, W/m·K.

3.2.9 β—fraction of pulse duration required to reach maximum intensity.

3.2.10 Δt_5—$T(5t_{1/2})/T(t_{1/2})$.

3.2.11 Δt_{10}—$T(10t_{1/2})/T(t_{1/2})$.

3.3 *Description of Subscripts Specific to This Standard:*

3.3.1 o—ambient.

3.3.2 s—specimen.

3.3.3 T—thermocouple.

3.3.4 x—percent rise.

3.3.5 C—Cowan.

3.3.6 R—ratio.

3.3.7 m—maximum.

3.3.8 t—time.

4. Summary of Test Method

4.1 A small, thin disc specimen is subjected to a high-intensity short duration radiant energy pulse. The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear face temperature rise (thermogram) is recorded. The thermal diffusivity value is calculated from the specimen thickness and the time required for the rear face temperature rise to reach certain percentages of its maximum value (Fig. 1). When the thermal diffusivity of the sample is to be determined over a temperature range, the measurement must be repeated at each temperature of interest. This test method is described in detail in a number of publications **(1, 2)**[4] and review articles **(3, 4, 5)**. A summary of the theory can be found in Appendix X5.

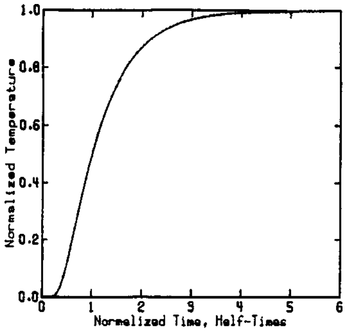

FIG. 1 Characteristic Thermogram for the Flash Method

5. Significance and Use

5.1 Thermal diffusivity is an important property, required for such purposes as design applications under transient heat flow conditions, determination of safe operating temperature, process control, and quality assurance.

5.2 The flash method is used to measure values of thermal diffusivity, α, of a wide range of solid materials. It is particularly advantageous because of simple specimen geometry, small specimen size requirements, rapidity of measurement and ease of handling, with a single apparatus, of materials having a wide range of thermal diffusivity values over a large temperature range.

5.3 Under certain strict conditions, specific heat capacity of a homogeneous isotropic opaque solid sample can be determined when the method is used in a quantitative fashion (see Appendix X4).

5.4 Thermal diffusivity results, together with specific heat capacity (C_p) and density (ρ) values, can be used in many cases to derive thermal conductivity (λ), according to the relationship:

$$\lambda = \alpha\, C_p\, \rho. \tag{1}$$

6. Interferences

6.1 In principle, the thermal diffusivity is obtained from the thickness of the sample and from a characteristic time function describing the propagation of heat from the front surface of the sample to its back surface. The sources of uncertainties in the measurement are associated with the sample itself, the temperature measurements, the performance of the detector and of the data acquisition system, the data analysis and more specifically the finite pulse time effect, the nonuniform heating of the sample and the radiative heat losses. These sources of uncertainty can be considered systematic, and should be carefully considered for each experiment. Errors random in nature (noise, for example) can be best estimated by performing a large number of repeat experiments and comparing their results. The relative standard deviation of the obtained results is a good representation of the random component of the uncertainty associated with the measurement. Guidelines in performing a rigorous evaluation of these factors are given in **(31)**.

7. Apparatus

The essential components of the apparatus are shown in Fig. 2. These are the flash source, sample holder, environmental enclosure (optional), temperature response detector and recording device.

7.1 The flash source may be a pulse laser, a flash lamp, or other device capable to generate a short duration pulse of substantial energy. The duration of the energy flash should be less than 2 % of the time required for the rear face temperature rise to reach one half of its maximum value (see Fig. 1).

7.1.1 The pulse hitting the sample's surface must be spatially uniform in intensity. Most pulse lasers exhibit hot spots and a substantially higher intensity in the center region of the beam than in the periphery. For this reason, systems using unmodified beams directly from a pulse laser should use beams somewhat larger in diameter than the largest diameter of the samples to be tested. The use of an optical fiber between the laser and the sample improves substantially uniformity of the beam (up to 95 %). Since this method produces almost no edge effects, a larger portion of the energy can be directed to the sample than for natural beam lasers.

7.1.2 Most commonly used lasers are: ruby (visible red),

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[4] The boldface numbers given in parentheses refer to a list of references at the end of the text.

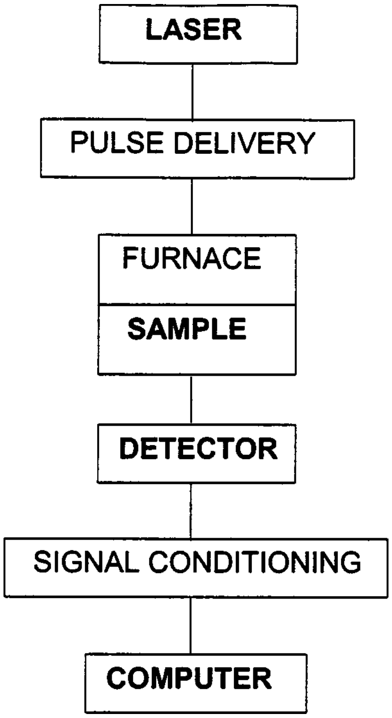

FIG. 2 Block Diagram of a Flash System

Nd: glass, and Nd: YAG (near infrared); however, other types of lasers may be used. In some instances properly engineered Xenon flash sources can provide comparable performance for all but the shortest rise times. Xenon flash sources, when properly focused, provide a lower cost and lower maintenance alternative to lasers for many applications.

7.2 An environmental control chamber is required for measurements above and below room temperature. This chamber must be gas or vacuum tight if operation in protective atmosphere is desired. The enclosure shall be fitted with a window, which has to be transparent to the flash source. A second window is required if optical detection of the rear face temperature rise is used. In such cases it is recommended that the optical detector be shielded from direct exposure to the energy beam with the use of appropriate filter(s).

7.3 The furnace or cryostat should be loosely coupled (thermally) to the specimen support and shall be capable of maintaining the specimen temperature constant within 4 % of the maximum temperature rise over a time period equal to five halves of the maximum rise time. The furnace may be horizontal or vertical. The specimen support shall also be loosely coupled thermally to the specimen. Specimen supports may be constructed to house one sample or several samples at a time, with the latter providing substantial improvements in data and testing speed.

7.4 The detector can be a thermocouple (see Appendix X2), infrared detector, optical pyrometer, or any other means that can provide a linear electrical output proportional to a small temperature rise. It shall be capable of detecting 0.05 K change above the sample's initial temperature. The detector and its associated amplifier must have a response time substantially smaller than 2 % of the half time value. When intrinsic thermocouples are used, the same response requirements shall apply. Electronic filters, if used, shall be verified not to distort the shape of the thermogram. Several precautions are required when using optical temperature sensing. The sensor must be focused on the center of the back surface of the sample. It also must be protected from the energy beam, to prevent damage or saturation. When the specimen is housed in a furnace, the energy beam may bounce or shine past the edges and enter the detector. To avoid this, proper shielding is necessary. For protection against lasers, dielectric spike filters that are opaque at the selected wavelength are very useful. The viewing window and any focusing lenses must not absorb appreciably the radiation in the wavelength region of the detector. This is particularly important for infrared detectors, and means should be provided to ensure that during high temperature measurements all window surfaces are monitored and kept free of deposits, which might lead to absorption of energy. Such build-ups can lead to loss of signal intensity and may cause non-uniform specimen heating from the energy source.

7.5 The signal conditioner includes the electronic circuit to bias out the ambient temperature reading, spike filters, amplifiers and analog-to-digital converters.

7.6 *Data Recording*

7.6.1 The data collection system must be of adequate speed to ensure that time resolution in determining half of the maximum temperature rise on the thermogram is at least 1 %, for the fastest thermogram for which the system is qualified.

7.6.2 The recorded signal must contain information that enables the precise definition of the starting time of the energy pulse.

7.6.2.1 If no other means are available, the inevitable spike caused by the trigger pulse (for a laser of flash lamp) may be used. This, however, is considered marginal, as it uses the beginning of the capacitor discharge as "time zero."

7.6.2.2 More accurate results are obtained if the center of gravity for the energy pulse is used as "time zero." This can be determined only with actual recording of the pulse shape and derivation of the point of start for each pulse. This also takes into account the varying energy of each pulse whether controlled or uncontrolled.

7.6.3 It is desirable to employ a data recording system that is capable of preprogrammed multiple speed recording within a single time period. This enables high-resolution (fast) recording prior to and during the rising portion of the thermogram, and lower resolution (slow) recording of the prolonged cool-down of the sample. (The cool-down portion of the thermogram is used for heat loss corrections — see later sections.)

7.6.4 In case the recording device does not have accurate built-in training (such as for digital systems), the timing accuracy must be verified periodically to ensure that the half of the maximum rise time is measured within 2 % for the fastest expected signal.

7.7 It is practical to incorporate an alignment device such as a He-Ne laser or a laser diode into the system, to aid with verifying proper positioning of the sample. The alignment beam must be at all times co-linear with the energy pulse path within 1 %.

7.8 An aperture must be provided in close proximity of the sample, to ensure that no portion of the energy beam will shine by the sample. It is desirable to keep this aperture's diameter approximately 95 % of the sample diameter. Providing a too small aperture will cause uneven sample heating and promote bi-axial heat-flow within the sample. A too large aperture will defeat the purpose. Systems with pin type sample suspensions are especially in need of accurate alignment and effective aperture size.

7.9 Measurement of sample temperature is to be done by accepted means, such as calibrated thermocouple, optical pyrometer, platinum RTD, etc. whichever is appropriate for the temperature range. In all cases, such a device must be in intimate contact with or trained on the sample holder, in close proximity of the sample. Touching the sample with thermocouples is not recommended. Embedding thermocouples into the sample is not acceptable.

7.10 The temperature controller and/or programmer are to bring the specimen to the temperatures of interest. While it is desirable to perform the measurements at exact temperatures, in most cases it is not necessary to exactly settle at those temperatures when the testing program covers a temperature range. It is uneconomical time-wise to try to reach an exact temperature when the thermal diffusivity is expected to behave monotonically in the range. In cases when the sample is expected to undergo internal transformations during the test, the temperatures of interest must be closely observed.

8. Test Specimen

8.1 The usual specimen is a thin circular disc with a front surface area less than that of the energy beam. Typically, specimens are 6 to 18 mm in diameter. The optimum thickness depends upon the magnitude of the estimated thermal diffusivity, and should be chosen so that the time to reach the maximum temperature falls within the 40 to 200 ms range. Thinner specimens are desired at higher temperatures to minimize heat loss corrections; however, specimens should always be thick enough to be representative of the test material. Typically, thicknesses are in the 1 to 6 mm range. Since the thermal diffusivity is proportional to the square of the thickness, it may be desirable to use different thicknesses in different temperature ranges. In general, one thickness will be far from optimum for measurements at both cryogenic and high temperature.

8.2 Inappropriately selected sample thickness will not only cause unnecessary frustration for the experimenter, but also can be a major source of error in the measurement. As a general guideline, one can start with 2 to 3 mm thick samples, and later change them based on the information obtained from the thermogram. (An overly thick sample can totally extinguish the signal.)

8.3 Samples must be prepared with faces flat and parallel within 0.5 % of their thickness. Non-uniformity of either surface (craters, scratches, markings) is not acceptable to have, as it will severely affect data.

9. Calibration and Verification

9.1 Calibrate the micrometer used to measure the specimen thickness, so that the thickness measurements are accurate to within 0.2 %.

9.2 The Flash Method is an absolute (primary) method by itself, therefore it requires no calibration. However, actual execution of the measurement itself is subject to random and systematic errors. It is therefore important to verify the performance of a device, to establish the extent these errors may affect the data generated. This can be accomplished by testing one or several materials whose thermal diffusivity is well known. While most materials used are not true certified standards, they are generally accepted industry-wide with the best available literature data (see Appendix X3).

9.2.1 It must be emphasized that the use of reference materials to establish validity of the data on unknown materials has often led to unwarranted statements on accuracy. The use of references is only valid when the properties of the reference (including half times and thermal diffusivity values) are closely similar to those of the unknown sample, and the temperature-rise curves are determined in an identical manner for the reference and unknown.

9.2.2 One important check of the validity of data (in addition to the comparison of the rise curve with the theoretical model), when corrections have been applied, is to vary the specimen thickness. Since the half times vary as L^2, decreasing the specimen thickness by one-half should decrease the half time to one-fourth of its original value. Thus, if one obtains the same thermal diffusivity value with representative specimens from the same material of significantly different thicknesses, the results can be assumed valid.

10. Procedure

NOTE 1—It is a good practice to apply a very thin, uniform graphite or other high emissivity coating on both faces of the sample to be tested, prior to performing the measurements. The coating may be applied by spraying, painting, sputtering, etc. This will improve the capability of the sample to absorb the energy applied, especially in case of highly reflective materials.

10.1 For commercially produced systems, follow manufacturer's instructions.

10.2 As a minimum, any system must ensure the following, either by design or by adjustment procedure:

10.2.1 Verification of sample concentricity with energy beam when properly mounted in holder.

10.2.2 Verification of aperture and energy beam coverage on sample.

10.2.3 Permanent alignment features for detector or means to properly align detector on center of rear surface.

10.2.4 Safety interlocks in case of lasers to prevent the escape of laser beam directly or reflections thereof.

10.3 The testing procedure must contain the following functions:

10.3.1 Determine and record the specimen thickness.

10.3.2 Mount the specimen in its holder.

10.3.3 Establish vacuum or inert gas environment in the chamber if necessary.

10.3.4 Determine specimen temperature unless the system will do it automatically.

10.3.5 Especially at low temperatures, use the lowest level of power for the energy pulse able to generate a measurable temperature rise, in order to ensure that the detector functions within its linear range.

10.3.6 After the pulse delivery, monitor the raw or processed thermogram to establish in-range performance. In case of multiple sample testing, it is advisable (for time economy) to sequentially test samples at the same temperature before doing replicate tests.

10.3.7 In all cases, the temperature stability prior and during a test must be verified either manually or automatically to be within specifications. Testing on a ramp is not recommended.

10.3.8 Determine the specimen ambient temperature and collect the base line, transient-rise and cooling data, and analyze the results according to Section 11.

10.3.9 Change or program the specimen ambient temperature as desired and repeat the data collection process to obtain measurements at each temperature.

10.3.10 If required, repeat the measurements at each temperature on the specimen's cooling or on its re-heating over the same cycle.

11. Calculation

11.1 First determine the baseline and maximum rise to give the temperature difference, ΔT_{max}. Determine the time required from the initiation of the pulse for the rear face temperature to reach $\Delta T_{1/2}$. This is the half time, $t_{1/2}$. Calculate the thermal diffusivity, α, from the specimen thickness, *L*, in m and the half time $t_{1/2}$ in s, as follows:

$$\alpha = 0.13879\, L^2/t_{1/2} \tag{2}$$

Check the validity of the experiment by calculating α at a minimum of two other points on the rise curve. The equation is as follows:

$$\alpha = k_x\, L^2/t_x \tag{3}$$

where:

t_x = the time required for the temperature rise to reach x percent of ΔT_{max}. Values of k_x are given in Table 1.

11.1.1 Ideally, the calculated values of α for different values of *x* should all be the same. If the values at 25, 50 and 75 % ΔT_{max} lie within ± 2 %, the overall accuracy is probably within ± 5 % at the half time. If the α values lie outside of this range, the response curve should be analyzed further to see if finite-pulse time, radiation heat loss or non-uniform heating effects are present.

11.1.2 Thermal radiation heat loss effects are most readily determined from the temperature of the specimen and the rear-face temperature response after $4t_{1/2}$. The recommended procedure is to plot the experimental values of $\Delta T/\Delta T_{max}$ versus $t/t_{1/2}$ along with the values for the theoretical model. Some numbers for the theoretical model are given in Table 2.

11.1.3 A plot of the normalized experimental data and the theoretical model can be prepared readily on line with a computer-based data acquisition system or by preparing graphs using the tabulated values of $\Delta T/\Delta T_{max}$ and $t/t_{1/2}$ and plotting the corresponding experimental data at several percent levels of the rise. All normalized experimental curves must pass through $\Delta T/\Delta T_{max} = 0.5$ and $t/t_{1/2} = 1.0$. Therefore, calculations including the 25 to 35 % and 66.67 to 80 % ranges are required to compare the experimental data with the theoretical curve.

TABLE 1 Values of the Constant k_x for Various Percent Rises

x(%)	k_x	x(%)	k_x
10	0.066108	60	0.162236
20	0.084251	66.67	0.181067
25	0.092725	70	0.191874
30	0.101213	75	0.210493
33.33	0.106976	80	0.233200
40	0.118960	90	0.303520
50	...	...	...

TABLE 2 Values of Normalized Temperature Versus Time for Theoretical Model

$\Delta T/\Delta T_{max}$	$t/t_{1/2}$	$\Delta T/\Delta T_{max}$	$t/t_{1/2}$
0	0	0.7555	1.5331
0.0117	0.2920	0.7787	1.6061
0.1248	0.5110	0.7997	1.6791
0.1814	0.5840	0.8187	1.7521
0.2409	0.6570	0.8359	1.8251
0.3006	0.7300	0.8515	1.8981
0.3587	0.8030	0.8656	1.9711
0.4140	0.8760	0.8900	2.1171
0.4660	0.9490	0.9099	2.2631
0.5000	1.0000	0.9262	2.4091
0.5587	1.0951	0.9454	2.6281
0.5995	1.1681	0.9669	2.9931
0.6369	1.2411	0.9865	3.6502
0.6709	1.3141	0.9950	4.3802
0.7019	1.3871	0.9982	5.1102
0.7300	1.4601	...	...

11.1.4 Examples of the normalized plots for experiments that approximate the ideal case, in which there is a finite pulse time effect and in which there are radiation heat losses, are shown in Figs. 3 and 4, and Fig. 5, respectively. Various procedures for correcting for these effects are also given in Refs. **(4, 15, 16, 17, 18, 19, 25, 26)** and specific examples are given in 11.2 and 11.3. The corrections can be minimized by the proper selection of specimen thickness. The finite pulse time effect decreases as the thickness is increased while heat losses decrease as the thickness is reduced.

11.1.5 Non-uniform heating effects also cause deviations of the reduced experimental curve from the model because of two-dimensional heat flow. Since there are a variety of non-uniform heating cases, there are a variety of deviations. Hot center cases approximate the radiation heat loss example. Cold center cases result in the rear face temperature continuing to rise significantly after $4t_{1/2}$. Non-uniform heating may arise

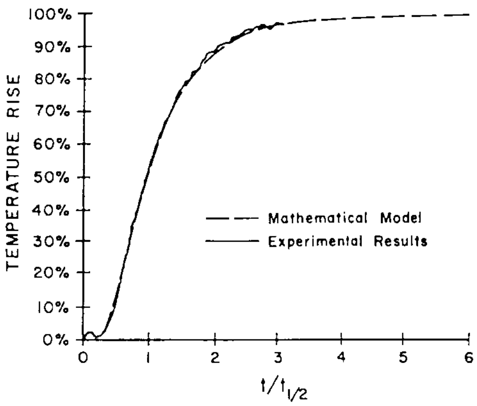

FIG. 3 Comparison of Non-dimensionalized Temperature Response Curve to Mathematical Model

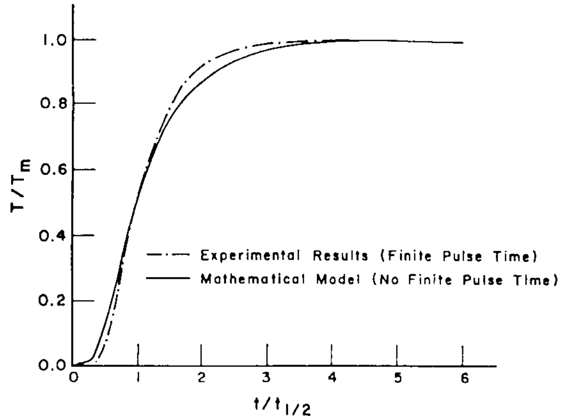

FIG. 4 Normalized Rear Face Temperature Rise: Comparison of Mathematical Model (No Finite Pulse Time Effect) to Experimental Values with Finite Pulse Time

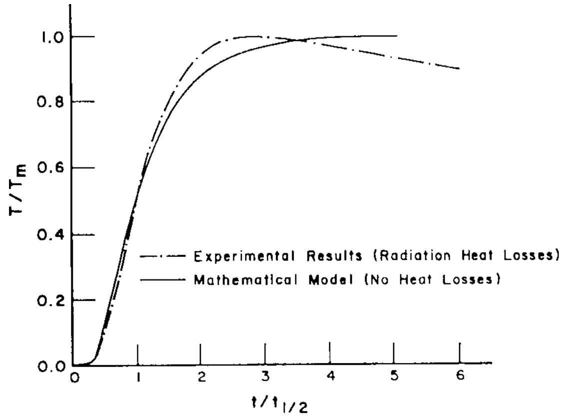

FIG. 5 Normalized Rear Face Temperature Rise: Comparison of Mathematical Model (No Heat Loss) to Experimental Values with Radiation Heat Losses

from the nature of the energy pulse or by non-uniform absorption on the front surface of the specimen. The former case must be eliminated by altering the energy source while the latter may be eliminated by adding an absorbing layer and using two-layer mathematics **(4, 13)**.

11.2 Finite pulse time effects usually can be corrected for using the equation:

$$\alpha = K_1 L^2 / (K_2 t_x - \tau). \tag{4}$$

For this to be valid, the energy pulse must be representable by a triangle of duration τ and time to maximum intensity of βτ as shown in Fig. 6. The pulse shape of the energy pulse for the laser should be determined using an optical detector that can detect the laser pulse as opposed to the flash lamp pulse. From this pulse shape β and τ are obtained. Values of the two constants K_1 and K_2 for various values of β are given in Table 3 for correcting $\alpha_{0.5}$.

11.3 Heat loss corrections should be based using both Clark and Taylor rise curve data **(25)** and Cowan cooling curve data **(26)**. These corrections are affected by non-uniform heating effects. Cooling curve corrections are affected by conduction losses to the holders in addition to the radiation losses from the surfaces. Thus, the errors in the correction procedures are affected by different phenomena and a comparison of thermal diffusivity values corrected by the two procedures is useful in determining the presence or absence of these phenomena.

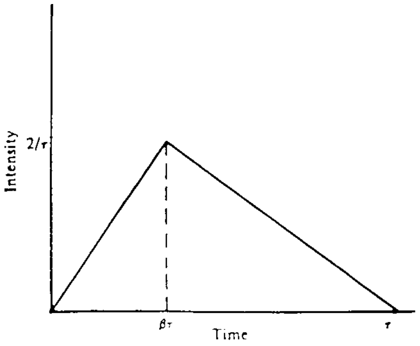

FIG. 6 Laser Pulse Shape

TABLE 3 Finite Pulse Time Factors

b	K_1	K_2
0.15	0.34844	2.5106
0.28	0.31550	2.2730
0.29	0.31110	2.2454
0.30	0.30648	2.2375
0.50	0.27057	1.9496

11.3.1 To use the Cowan cooling curve corrections, determine the ratio of the net rise time values at times that are five and ten times the experimental half time value to the net rise at the half time value. These ratios are designated as Δt_5 and Δt_{10}. If there are no heat losses $\Delta t_5 = \Delta t_{10} = 2.0$. The correction factor (K_C) for the five and ten half time cases are calculated from the polynominal fits:

$$K_C = A + B(\Delta t) + C(\Delta t)^2 + D(\Delta t)^3 + E(\Delta t)^4 + F(\Delta t)^5 + G(\Delta t)^6 + H(\Delta t)^7 \tag{5}$$

where:

values for the coefficients *A* through *H* are given in Table 4. Corrected values for diffusivity are calculated from the following relation:

$$\alpha_{corrected} = \alpha_{0.5} K_C / 0.13885 \tag{6}$$

where:

$\alpha_{0.5}$ = the uncorrected thermal diffusivity value calculated using the experimental half time.

11.3.2 Heat loss corrections based on the Clark and Taylor rise curve data also use ratio techniques **(24)**. For the $t_{0.75}/t_{0.25}$ ratio, that is, the time to reach 75 % of the maximum divided by the time to reach 25 % of the maximum, the ideal value is 2.272. Determine this ratio from the experimental data. Then calculate the correction factor (K_R) from the following equation:

TABLE 4 Coefficients for Cowan Corrections

Coefficients	Five Half Times	Ten Half Times
A	−0.1037162	0.054825246
B	1.239040	0.16697761
C	−3.974433	−0.28603437
D	6.888738	0.28356337
E	−6.804883	−0.13403286
F	3.856663	0.024077586
G	−1.167799	0.0
H	0.1465332	0.0

$$K_R = -0.3461467 + 0.361578\,(t_{0.75}/t_{0.25}) - 0.06520543\,(t_{0.75}/t_{0.25})^2 \quad (7)$$

The corrected value for the diffusivity at the half time is $\alpha_{corrected} = \alpha_{0.5}\, K_R/0.13885$. Corrections based on many other ratios can also be used.

12. Report

12.1 As a minimum, the report shall contain the following information:

12.1.1 Identification of the sample (material) and previous history;

12.1.2 Sample thickness (m);

12.1.3 Temperature (°C);

12.1.4 Calculated value of thermal diffusivity at x = 50 %, m^2/s, at the reported temperature;

12.1.5 Statements concerning calculated values near x = 25 and 75 % as well as x = 50 %, or a comparison of the reduced experimental curve to the model, at each temperature;

12.1.6 Statements concerning the results of repeat measurements at each temperature;

12.1.7 Statement as to whether or not the data was corrected for thermal expansion. If this correction was made, the thermal expansion values used must be reported;

12.1.8 Discussion of errors and correction procedures that were used for heat losses and finite pulse time effects;

12.1.9 Environmental surroundings of the specimen;

12.1.10 Statements of conformance with requirements of this standard.

12.2 Additionally, it is beneficial to report:

12.2.1 Statement that the response time of the detector, including the associated electronics was adequately checked, and the method used;

12.2.2 Energy pulse source;

12.2.3 Statement of the beam uniformity check, or methods employed to eliminate the need for any;

12.2.4 Type of temperature rise detector.

13. Precision and Bias

13.1 A number of national and international round robins have shown that a measurement precision of ± 5 % can be attained for thermal diffusivity of a variety of materials. The results of several of these programs are detailed in a separate report on file with ASTM. No evidence of bias has been noted for opaque materials. Generally the values were obtained using simple data acquisitions and analysis. It has been shown that the accuracy can be significantly improved using more sophisticated data acquisition and data analysis.

13.2 The above precision levels do not imply that the specific heat capacity and thermal conductivity of the specimen can be derived to the same levels from thermal diffusivity measurements, since such derivations require input of values for other parameters.

14. Keywords

14.1 flash method; infrared detectors; intrinsic thermocouples; POCO graphite; specific heat capacity; thermal conductivity; thermal diffusivity; transient temperature measurements

APPENDIXES

(Nonmandatory Information)

X1. TESTING NON-IDEAL SAMPLES

While this test method was developed for and applied originally to homogeneous opaque solids, it can be extended under appropriate conditions to a wide variety of materials and situations. These include heterogeneous specimens of dispersed composites **(6)**, layered structures **(7, 8)** translucent materials, liquids and coatings **(9, 10)** and the measurement of contact conductance and resistance **(11, 12)**.

X1.1 Translucent or transparent samples must be made opaque to the energy pulse by depositing a very thin continuous layer of opaque material such as a metal film on the two surfaces. Doing so, care must be exercised to select a material that will withstand the temperature to which the sample will be subjected, and will not crack or peel off due to excessively different coefficients of thermal expansion. A cracked layer will allow partial penetration of the pulse into the interior of the sample and will distort the rear face thermogram. Peeled coatings will cause localized heating, excessive attenuation, and often total extinction of meaningful signals.

X1.1.1 Most frequently used coatings are gold, platinum, aluminum, nickel, and silver.

X1.1.2 A thin sprayed layer of powder, such as graphite, is usually not dense enough to properly block the energy by itself.

X1.1.3 High reflectivity coatings, such as gold or platinum, require a second coat of graphite on both faces of the sample, to ensure that the energy pulse will be absorbed on the surface.

X1.2 Testing liquids and molten metals by this method is advantageous because the speed with which the test proceeds precludes heat transfer by convection. The sample is normally enclosed in a container that must have provisions to maintain a known sample thickness throughout the test, allow for escape of the excess liquid upon heating, and transmit the energy pulse to the front face as well as the temperature signal from rear face, with minimal attenuation.

X1.2.1 When possible to use transparent top and bottom windows for the containment capsule, the liquid sample is evaluated as if it were solid.

X1.2.2 When transparent windows are not feasible to use due to temperature limitations or materials interaction, a suitable opaque material is used instead. In this case, the analysis follows the three layer calculations.

X1.3 Testing multi-layer samples is possible in most cases when the ratios of thermal diffusivity and the overall thickness are within the operating limits of the instrument. Most commonly, the analysis for these cases **(6, 7)** also contains the necessary inclusion of heat loss and other corrections, without which its' utility is diminished.

X2. THERMOCOUPLE TYPE DETECTORS

Under certain conditions, it is advantageous to use thermocouples for signal detection. Most frequently they are used in cases where optical sensors are not practical, such as near and below ambient temperatures. There are two methods in use: intrinsic thermocouple and beaded thermocouple.

X2.1 For intrinsic thermocouples, the two legs of the thermocouple are not joined together in a bead, but are individually making contact with the sample, thereby having the sample itself part of the circuit. Thermoelectric EMF is generated at the points of contact for both legs. Since these points of contact are on the surface of the specimen, the net EMF of the couple closely reflects the temperature of the surface.

X2.1.1 Intrinsic thermocouples can be used only with electrically conductive samples or with non-conductive samples covered with a very thin conductive layer (vacuum deposited metal, conductive paint, etc.). The thermocouple wires are often formed into a sharp pin, which is then pressed against the sample or this conductive layer.

X2.1.2 The term "thermocouple" in this procedure is meant to also include other forms of thermoelectric materials besides conventional thermocouple alloys, such as semiconductors, which can provide sufficient thermoelectric EMF for the purpose.

X2.2 Beaded thermocouples are sometimes used when intrinsic couples are not practical. In these cases, special care must be exercised to ensure that the beaded couple truly reflects the response of the back surface of the sample.

X2.3 Intrinsic thermocouples are preferred over beaded couples.

X2.4 The thermocouple material is not required to be calibrated, as the absolute magnitude of the measured signal is not relevant in the thermal diffusivity calculations.

X2.5 In the case of thermocouples, the response time (time to reach 95 % of steady-state value) can be defined **(20)** as follows:

$$t_{95} = \frac{25}{\pi} \cdot \frac{D^2_T}{\alpha_s} \cdot \frac{\lambda_T}{\lambda_S} \qquad \text{(X2.1)}$$

Thus, a small diameter thermocouple of low thermal conductivity material attached to a specimen of high thermal conductivity and high thermal diffusivity material yields the fastest response time. Eq X2.1 is misleading, in that it can postulate that the thermocouple response is a smooth rise. Actually, the response is a step change, followed by an exponential rise to the final value. This behavior is best represented by Eq X2.2:

$$\frac{T_t - T_0}{T_\infty - T_0} = 1 - (1 - a) \cdot e^{a^2} \cdot t^* \cdot Erfc(a \cdot t^*) \qquad \text{(X2.2)}$$

where:

T_0 and T_∞ are shown in Fig. X2.1, t^* is dimensionless time ($t^* = 4\alpha_s \cdot t/D_T{}^2$), and a is approximated by $1/(1 + 0.667\ \lambda_T/\lambda_s)$. In order to obtain the fastest response, small diameter thermocouple wire of an alloy having a low thermal conductivity attached to a substrate of high thermal diffusivity should be used. For example, a 25 µm constantan wire on a copper substrate requires 3 µs to reach 95 % of steady-state. However, for the converse of this example, for example 25 µm copper wire on a constantan substrate, it is found that 15 ms are required to reach 95 % of the steady-state. This is 5000 times slower than in the first example. Thus, the proper selection of materials, based upon their thermal properties and geometries, is essential for accurate measurement of transient responses using thermocouples **(21)**.

Eq X2.1 and X2.2 relate to the minimum response time possible for a thermocouple. Proper attachment of the thermocouple is important since, if the thermocouple is attached poorly to the specimen, the effective response time can be much longer. The preferred method for electrical conducting materials is to spot-weld intrinsic thermocouples, that is, non-beaded couples where each leg is independently attached to the specimen about 1 mm apart. For electrical insulators, where spot welding is not feasible, it may be possible to spring-load the thermocouple against the back surface. For materials with low thermal diffusivity values, it may be preferred to spot-weld thermocouples onto a thin high thermal conductivity metallic sheet and spring-load or paste this sheet onto the specimen. Metal-epoxy and graphite pastes have been used successfully to bond layers together. This eliminates the problem of using thermocouples of relatively high thermal diffusivity to measure specimens of materials of low thermal diffusivity, that can lead to very large response times (see Eq X2.1).

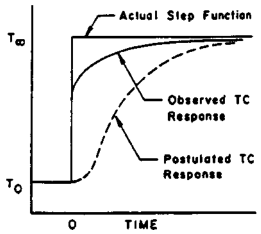

FIG. X2.1 Thermocouple Response Characteristics

X3. REFERENCE MATERIALS

As of the time of the issue of this Standard, there are no Standard Reference Materials (SRM) available from the National Institute of Standards and Technology (NIST) for thermal diffusivity. Also there are no references available from other standards institutes from abroad. However, a large amount of data is available in the literature on a number of industry-accepted reference materials that have been used for verification purposes.

X3.1 *Thermal Conductivity SRM*—One may consider the use of related standards such as thermal conductivity SRM's with derived values for thermal diffusivity. Available presently:

X3.1.1 *Electrolytic Iron (Research Materials 8420 and 8421)*. This is very similar to what the Armco Iron was, which was much tested but is no longer made, and therefore the literature data is quite applicable **(22, 23)**. Furthermore, it is quite proper to calculate thermal diffusivity for this material using the method described in Appendix X4.

X3.1.2 *POCO AXM-5QA Graphite (Research Materials 8424, 8425 and 8426)*. Although this material is very widely used, it requires extreme care. Due to material variability (density and electrical resistivity) across the mother blocks from which the SRM pieces were cut, equally large variations in thermal properties are present. For this reason, the data accompanying each piece must be modified before being used, to reflect these variations. Additionally, the temperature dependence of thermal diffusivity for the material is very sharp near ambient, and therefore its utility in this region is questionable. (A very small error in the sample temperature will result in a disproportionally large change in thermal diffusivity.)

X3.2 Other materials that have been extensively studied are: OFHC Copper, HP Copper, Pyroceram, HP Aluminum, Nickel, and Glassy Carbon. A valuable summary and data bank of thermal diffusivity values found in the literature that also provides recommended values for a wide range of materials, is also available **(24)**.

X3.3 It must be emphasized that the use of reference materials to establish the validity of the data on unknown materials has often led to unwarranted statements on accuracy. The use of references is only valid when the properties of the reference (including half times and thermal diffusivity values) are closely similar to those of the unknown, and the temperature-rise curves are determined in an identical manner for the reference and unknown.

X4. MEASUREMENT OF SPECIFIC HEAT CAPACITY AND CALCULATION OF THERMAL CONDUCTIVITY

X4.1 The fundamental relationship between thermal diffusivity (α), thermal conductivity (λ), specific heat capacity (C_p), and density (ρ),

$$\alpha = \frac{\lambda}{C_p \cdot \rho} \tag{X4.1}$$

allows the calculation of thermal conductivity, a much sought after property, with the knowledge of the other properties. A method was developed where the specific heat capacity of a sample is determined when the thermal diffusivity test is performed in a quantitative fashion. Although this is a very attractive extension of the method, one must exercise extreme caution in performing it, as the opportunity for errors abounds. In the course of an ordinary thermal diffusivity test, the amount of energy is important only to the extent that it will generate a sufficient rear face signal. For operating in a calorimetric mode, the energy level must be known closely, controllable and repeatable. Approximating adiabatic conditions, fortunately the laser pulse and the detector can be calibrated in unison when a sample of known specific heat capacity is tested. The measurement will yield thermal diffusivity, and also a relative measure of energy expressed in terms of the absolute value of the maximum attained temperature. By testing an unknown sample after this "calibration", the specific heat capacity can be calculated from its maximum attained temperature, relative to the one obtained for the standard. There are several conditions that must be satisfied in order for this process to be valid:

X4.1.1 The energy source must be able to reproduce the energy of a pulse based on the power defining parameter (charge voltage for lasers, for example) over a period of time.

X4.1.2 The detector must maintain its sensitivity over a period of time without drift, gain change, and within a linear response range.

X4.1.3 The reference sample and the unknown sample must be very similar in size, proportions, emissivity, and opacity, to approximate adiabatic behavior to the same extent. Both the reference and the unknown sample should be coated with a thin uniform graphite layer, to ensure that the emissivity of the two materials is the same.

X4.1.4 Both reference and unknown sample must be homogeneous and isotropic, as Eq X4.1 only applies for those materials. Heterogeneous and anisotropic materials will frequently produce erroneous data. The process is not purely calorimetric, since the maximum temperature rise is derived from the signal provided largely by the components with the highest thermal diffusivity, while the internal equilibration may take place after that point in time. For this reason, this method tends to give erroneous results for specific heat capacity for materials with large anisotropy (typically composites with an ordered directional structure) and for mixtures of components with greatly differing thermal diffusivity.

X4.1.5 The reference and the unknown must be tested very close to each other, both temporally (preferably only minutes apart) and thermally (strictly at the same temperature, in the same environment).

X4.1.6 Verification of the process is to be done using the same sample for unknown as was used for calibration, in the exact time interval (delay) as testing is normally conducted. The same test must be performed with three different thicknesses. The combined data from these three tests at each temperature gives a good measure of implied accuracy.

X4.1.7 This being a differential measurement, it is highly desirable to have both reference and unknown tested side-by-side and with very small time intervals in between. It is also desirable to test standard/sample/standard, to minimize errors from pulse energy variations.

X4.2 The sample's density may be calculated from results of weight measurements and computed volume. It is appropriate to calculate the density at each temperature from the room temperature density, using thermal expansion data. Consult ASTM E 228 for details.

X4.3 Thermal conductivity may be calculated using Eq X4.1, from the measured values of thermal diffusivity, specific heat capacity and density.

X4.3.1 When measured values of specific heat capacity are used, the constrains listed under X4.1.1-X4.1.7 also apply to the resultant thermal conductivity.

X4.3.2 It is permissible to use specific heat capacity and density data from other sources than the measurements above.

X4.4 Reporting specific heat capacity or thermal conductivity obtained in this manner must be accompanied by:

X4.4.1 Accuracy statement determined as given in X4.1.6;

X4.4.2 The time elapsed between calibration and test pulses;

X4.4.3 Calibration sample used.

X5. THEORY

X5.1 *The Ideal Case*—The physical model of the pulse method is founded on the thermal behavior of an adiabatic (insulated) slab of material, initially at constant temperature, whose one side has been subjected to a short pulse of energy. The model assumes:

- one dimensional heat flow;
- no heat losses from the slab's surfaces;
- uniform pulse absorption at the front surface;
- infinitesimally short pulse duration;
- absorption of the pulse energy in a very thin layer;
- homogeneity and isotropy of the slab material;
- property invariance with temperature within experimental conditions.

In deriving the mathematical expression from which the thermal diffusivity is calculated, Parker **(1)** starts from the equation of the temperature distribution within a thermally insulated solid of uniform thickness L, as given by Carslaw and Jaeger **(27)**:

$$T(x,t) = \frac{1}{L}\int_0^L T(x,0)dx + \frac{2}{L}\sum_{n=1}^{\infty} exp\left(\frac{-n^2\pi^2\alpha t}{L^2}\right)\cdot cos\frac{n\,\pi x}{L}\int_0^L T(x,0)\,cos\frac{n\,\pi x}{L}dx \tag{X5.1}$$

where α is the thermal diffusivity of the material. If a pulse of radiant energy Q is instantaneously and uniformly absorbed in the small depth g at the front surface $x = 0$, the temperature distribution at that instant is given by

$$T(x,0) = \frac{Q}{\rho \cdot C \cdot g} \tag{X5.2}$$

for $0 < x < g$

and

$$T(x,0) = 0 \tag{X5.3}$$

for $g < x < L$.

With this initial condition, Eq X5.1 can be written as:

$$T(x,0) = \frac{Q}{\rho CL}\left[1 + 2\sum_{n=1}^{\infty} cos\frac{n\,\pi x}{L}\,\frac{sin\frac{n\,\pi g}{L}}{\frac{n\,\pi g}{L}}\cdot exp\left(\frac{-n^2\pi^2}{L^2}\alpha t\right)\right] \tag{X5.4}$$

where ρ is the density and C is the specific heat capacity of the material. In this application only a few terms will be needed, and since g is a very small number for opaque materials,

$$sin\,\frac{n\,\pi g}{L} \approx \frac{n\,\pi g}{L} \tag{X5.5}$$

At the rear surface, where $x = L$, the temperature history can be expressed by:

$$T(L,t) = \frac{Q}{\rho CL}\left[1 + 2\sum_{n=1}^{\infty}(-1)^n\cdot exp\left(\frac{-n^2\pi^2}{L}\alpha t\right)\right] \tag{X5.6}$$

Two dimensionless parameters, V and ω can be defined:

$$V(L,t) = \frac{T(L,t)}{T_M} \tag{X5.7}$$

$$\omega = \frac{\pi^2\,\alpha t}{L^2} \tag{X5.8}$$

T_M represents the maximum temperature at the rear surface. The combination of Eq X5.6-X5.8 yields:

$$V = 1 + 2\sum_{n=1}^{\infty}(-1)^n\cdot exp(-n^2\,\omega) \tag{X5.9}$$

When $V = 0.5$, $\omega = 1.38$, and therefore:

$$\alpha = \frac{1.38\cdot L^2}{\pi^2 t_{\frac{1}{2}}} \tag{X5.10}$$

or:

$$\alpha = 0.1388\,\frac{L^2}{t_{\frac{1}{2}}} \tag{X5.11}$$

where $t_{½}$ is the time required for the back surface to reach half of the maximum temperature rise. Schematically, a pulse experiment is represented in Fig. X5.1.

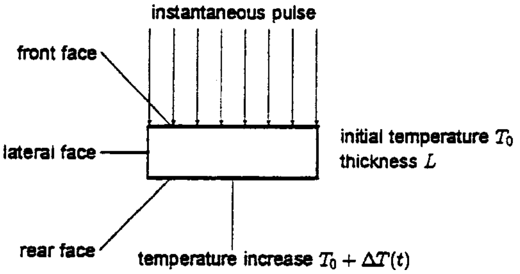

FIG. X5.1 Schematic of the Flash Method

As a result, a characteristic thermogram of the rear face is created (Fig. X5.2).

X5.2 *The Non-Ideal Case*—The inadequacy of the Parker solution became obvious almost immediately after its introduction, as nearly every one of these assumptions is violated to some extent during an experiment. So, gradually, investigators introduced various theories to describe the real process, and solutions describing corrections to counter the violation of each of the boundary conditions. The ideal correction would encompass all factors present, but to date, no such general correction has been developed. Instead, individual or paired corrections accounting for deviations were introduced. The result is that one can end up with an array of numbers that may vary substantially after using these corrections. This is understandable, as historically, each investigator has focused on one or another deviation from the ideal model, while assuming ideality and constancy of the others. This by itself is a substantial violation of principles, as in reality all parameters vary concurrently, in an extent dictated by the particular conditions of the experiment. Some situations may aggravate one condition, for example having a long pulse, others may introduce other deviations, such as excessive heat losses from the front face due to using very powerful pulses, etc. It is therefore incumbent upon the investigator to choose the most proper correction in harmony with the conditions of the experiment analyzed.

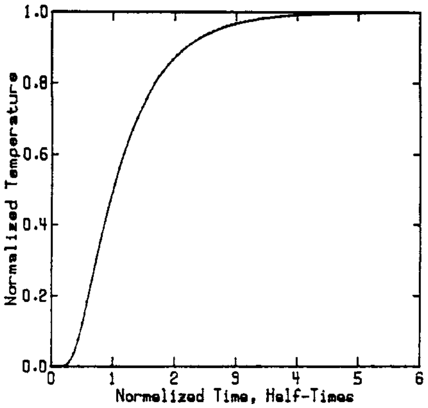

FIG. X5.2 Characteristic Thermogram for the Pulse Method

The finite pulse width effect, for example, occurs strongly when thin samples of high thermal diffusivity are tested **(2, 18, 19, 28, 29)**, while the radiative heat losses become dominant at high temperatures **(25)**, when testing thick samples. In contrast, nonuniform heating can occur during any thermal diffusivity experiment **(30)**. This can occur when a circular surface smaller than the sample itself is irradiated, or the flux density of the pulse varies from point-to-point over the sample's surface. For the same amount of absorbed energy, the dimensionless half-max time of the resulting thermogram at the center of the rear face of the specimen differs considerable from the one obtained with uniform irradiation. This effect can be reduced by increasing the ratio between the sample's thickness and its radius. The same result can be achieved by using a temperature measurement system, which automatically integrates the signal obtained from the rear face of the specimen.

It is a very difficult task to choose the best correction, and often not enough information is known about the equipment and the testing parameters to do it prudently. In principle, one must return to the original premise: the accuracy of the data depends on the agreement between the mathematical and experimental models. The purpose of applying corrections to the experimental data is to bring it to closer agreement with the ideal solution by accounting for the aberrations. In consequence, one can perform a series of corrections according to the various schemes developed over the years, and then study the results in relationship to the ideal solution.

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