



Standard Practice for Thermal Diffusivity by the Flash Method¹

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1. Scope

1.1 This practice covers practical details associated with the determination of the thermal diffusivity of primarily homogeneous isotropic solid materials. Thermal diffusivity values ranging from 10^{-7} to 10^{-3} m²/s are readily measurable by this from about 75 to 2800 K.

1.2 This practice is adjunct to Test Method E1461.

1.3 This practice 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.

1.4 This practice is intended to allow a wide variety of apparatus designs. It is not practical in a document 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. This practice provides guidelines for the construction principles, preferred embodiments and operating parameters for this type of instruments.

1.5 This practice 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 specimens. Since the magnitude of porosity, pore shapes, 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 The flash can be considered an absolute (or primary) method of measurement, since no reference materials are required. It is advisable to use only 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 been shown to produce data found to be useful in certain applications:

1.7.1 *Testing of Composite Materials*—When substantial non-homogeneity 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 composite materials, for example, may have heat flow patterns substantially different than uniaxial. In cases where the particle size of the composite phases is small compared to the specimen thickness (on the order of 1 to 25 % of thickness) and where the transient thermal response of the specimen appears homogenous when compared to the model, this method can produce accurate results for composite materials. Anisotropic materials can be measured by various techniques, as long as the directional thermal diffusivities (two dimensional or three dimensional) are mutually orthogonal and the measurement and specimen preparation produce heat flow only along one principle direction. Also, 2D and 3D models and either independent measurements in one or two directions, or simultaneous measurements of temperature response at different locations on the surface of the specimen, can be utilized.

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 specimen 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 the thermal properties of one of the layers are 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 standard. No other units of measurement are included in this 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.*

¹ This practice 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. Referenced Documents

2.1 ASTM Standards:²

E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

E1461 Test Method for Thermal Diffusivity by the Flash Method

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.

3.2 Description of Symbols and Units Specific to This Standard:

3.2.1 D —diameter, meters.

3.2.2 C_p —specific heat capacity, J/(kg·K).

3.2.3 k —constant depending on percent rise.

3.2.4 K —correction factors.

3.2.5 K_1 , K_2 —constants depending on β .

3.2.6 L —specimen thickness, m.

3.2.7 t —response time, s.

3.2.8 $t_{1/2}$ —half-rise time or time required for the rear face temperature rise to reach one half of its maximum value, s.

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

3.2.10 T —temperature, K.

3.2.11 α —thermal diffusivity, m²/s.

3.2.12 λ —thermal conductivity, (W/m·K).

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

3.2.14 ρ —density, kg/m³.

3.2.15 $\Delta t_5 = T(5t_{1/2})/T(t_{1/2})$.

3.2.16 $\Delta t_{10} = T(10t_{1/2})/T(t_{1/2})$.

3.2.17 $\Delta T_{\max}$ —temperature difference between baseline and maximum rise, K.

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 Practice

4.1 A small, thin disc specimen is subjected to a high-intensity short duration radiant energy pulse (Fig. 1). The

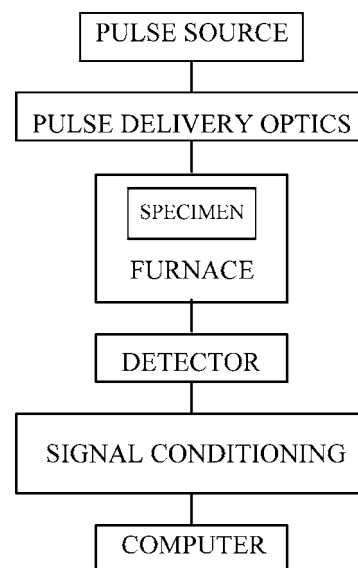


FIG. 1 Block Diagram of a Flash System

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. 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 is described in detail in a number of publications (1, 2)³ and review articles (3, 4, 5). A summary of the theory can be found in Test Method E1461, Appendix 1.

5. Significance and Use

5.1 Thermal diffusivity is an important property, required for such purposes under transient heat flow conditions, such as design applications, 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.

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 Test Method E1461, Appendix 1).

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

$$\lambda = \alpha C_p \rho. \quad (1)$$

6. Interferences

6.1 In principle, the thermal diffusivity is obtained from the thickness of the sample and from a characteristic time function

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

³ The boldface numbers given in parentheses refer to a list of references at the end of the text.

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 specimen and the heat losses (radiative and conductive). 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. The relative standard deviation of the obtained results is a good representation of the random component of the uncertainty associated with the measurement. Guidelines for performing a rigorous evaluation of these factors are given in (31).

7. Apparatus

The essential components of the apparatus are shown in Fig. 1. These are the flash source, specimen 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 pulse should be less than 2 % of the time required for the rear face temperature rise to reach one half of its maximum value, to keep the error due to finite pulse width less than 0.5 %, if pulse width correction (17, 18, 19) is not applied.

7.1.1 The pulse hitting the specimen'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 specimens to be tested. The use of an optical fiber between the laser and the specimen improves substantially the 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 specimen than from natural beam lasers.

7.1.2 Most commonly used lasers are: ruby (visible red), 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 a 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 specimen or several at a time, with the latter providing substantial improvements in data and testing speed.

7.4 The detector can be a thermocouple (see Appendix X1), 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 specimen's initial temperature. The detector and its associated amplifier must have a response time substantially smaller than 2 % of the half-rise 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 specimen. 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 acquisition system must be of an 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 or 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 Test Method E1461.)

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

7.9 Measurement of specimen 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 specimen holder, in close proximity of the specimen. Touching the specimen with thermocouples is not recommended. Embedding thermocouples into the specimen 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 specimen 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 10 to 12.5 mm in diameter, however, there is no fundamental limitation for using smaller or larger specimens. From a practical standard point, 12.5 mm was found to be ideal.

8.1.1 Specimens that are very small tend to provide small amounts of energy from the rear face, especially at low (< 400 °C) temperatures. For systems that have an appreciable distance from the specimen to the detector, such as most high temperature systems, this is a serious problem that should be avoided simply by using 10 mm diameter or larger specimens. Under all circumstances, one must not expect the same performance for sub-size specimens, under all conditions. Larger specimens on the other hand, may suffer from insufficient energy density, and produce more widely scattered data.

8.1.2 The optimum thickness depends upon the magnitude of the estimated thermal diffusivity, and should be chosen so that the time to reach half of the maximum temperature falls within the 10 to 1000 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.

8.1.3 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 temperatures.

8.2 Inappropriately selected specimen 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 specimens, and later change them based on the information obtained from the thermogram. (An overly thick specimen can totally extinguish the signal.)

8.3 Specimens must be prepared with faces flat and parallel within 0.5 % of their thickness, in order to keep the error in thermal diffusivity due to the measurement of average thickness, to less than 1 %. Non-uniformity of either surface (craters, scratches, markings) of significant depth compared to the specimen thickness should be avoided.

8.4 Proper surface preparation of specimens is imperative for obtaining reliable results.

8.4.1 Shiny surfaces, in large part, reflect light and, as a consequence, only a small amount of the total pulse energy is absorbed. To combat this, it is customary to deposit a very thin layer of highly energy absorbing (high emissivity) coating on the surface. Graphite has been found to work well, and is available in aerosol spray, paint, or colloidal suspension form. Other materials, such as boron nitride powder, have also been used.

NOTE 1—Material compatibility between the specimen and the coating must be investigated in all cases before a particular use, especially in high temperature applications. For example, graphite coating will react with an iron specimen, making the coating disappear at elevated temperatures, as well as potentially changing the composition of the specimen.

8.4.2 For transparent materials, it is customary to deposit a metal film (gold, platinum, silver, etc.) on both faces of the specimen, to make it opaque. Highly reflective materials are favored so that only a minute amount of the absorbed energy will be re-radiated by the other face of the metal film across the transparent medium, and the bulk of the energy will traverse it by heat conduction.

8.4.3 Since the highly reflective metal coating would not allow full absorption of the energy pulse, it is necessary to coat the specimen as per 8.4.1.

8.4.4 Conversely, since the shiny metal surface, due to its low emissivity, would produce a very weak optical target for obtaining the thermogram, the back face of the specimen has to be coated too as per 8.4.1.

8.4.5 In all cases, the combined effect of the coatings must be a negligible fraction of the total signal for any specimen, unless multi-layer analysis is applied.

8.4.6 Light sandblasting of specimen surfaces greatly enhances film adherence, and for some opaque reflective materials can provide sufficient pulse absorption and emissivity, especially at higher temperatures, where coatings may not be stable or may react with the material.

8.4.7 For specific heat capacity determinations, where two different surfaces are present (unknown and reference), proper and completely identical surface preparation for both specimen and reference is imperative. Since in this quantitative measurement the energy absorbed is fully controlled by the emissivity of the surface, both surfaces must present identical properties to the incoming energy pulse, to ensure a truly differential determination.

8.4.8 Encapsulated specimens should not be used for specific heat capacity tests, as the contribution of the capsule can not be mitigated via multi-layer calculations, and therefore the direct data will be in substantial error.

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 periodically, 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 (see Test Method [E1461](#), Appendix 3). While most materials used are not true certified standards, they are generally accepted industry-wide with the best available literature data.

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-rise times and thermal diffusivity values) are closely similar to those of the unknown specimen, 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-rise times vary as L^2 , decreasing the specimen thickness by one-half should decrease the half-rise 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

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 specimen concentricity with energy beam when properly mounted in holder.

10.2.2 Verification of aperture and energy beam coverage on specimen.

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 lasers are used, 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 specimen testing, it is advisable (for time economy) to sequentially test specimens at the same temperature (including replicate tests) before proceeding to the next test temperature.

10.3.7 In all cases, the temperature stability prior and during a test must be verified either manually or automatically to be less than 4 % of the maximum temperature rise. 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.

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 A pertinent computational model is presented in the Test Method [E1461](#).

11.2 This practice enlarges the presentation by concentrating on practical treatments, as related to testing non-ideal specimens.

12. Testing non-ideal specimens

12.1 While this practice 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).

12.2 Translucent or transparent specimens 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. In doing so, care must be exercised to select a material that will withstand the temperature to which the specimen 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 specimen and will distort the rear face thermogram. Peeled coatings will cause localized heating, excessive attenuation, and often total extinction of meaningful signals.

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

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

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

12.3 Testing liquids and molten metals by this method is advantageous because the speed with which the test proceeds precludes heat transfer by convection. The specimen is normally enclosed in a container that must have provisions to maintain a known specimen 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.

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

12.3.2 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.

12.4 Testing multi-layer specimens is possible in most cases when the ratio unknown to known layer diffusion time is favorable and the overall thickness and half-rise time 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.

13. Measurement of specific heat capacity and calculation of thermal conductivity

13.1 Eq 1 describes the relationship between thermal diffusivity (α), thermal conductivity (λ), specific heat capacity (C_p), and density (ρ), allowing the calculation of thermal conductivity, a much sought after property, with the knowledge of the other properties. A method was developed (1) where the specific heat capacity of a specimen 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 specimen 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 specimen 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:

13.1.1 The energy source must be able to reproduce within 5 % the energy of a pulse based on the power defining parameter (charge voltage for lasers, for example) over a period of time.

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

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

13.1.4 Both reference and unknown specimen must be homogeneous and isotropic, as Eq 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 diffusivities.

13.1.5 The reference and the unknown specimens 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).

13.1.6 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/specimen/standard, to minimize errors from pulse energy variations.

13.2 The specimen’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 Test Method E228 for details.

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

13.3.1 When measured values of specific heat capacity are used, the constraints listed under 13.1.1 – 13.1.6 also apply to the resultant thermal conductivity.

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

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

13.4.1 An accuracy statement.

13.4.2 The time elapsed between reference and test pulses.

13.4.3 Reference specimen used.

14. Thermal Conductivity Derivation

14.1 For this method, thermal conductivity is strictly a derived result, never a directly measured property. Thermal conductivity is calculated from Eq. (1) with the knowledge of

density (ρ). Either or both, specific heat capacity and thermal diffusivity may be obtained from direct measurement by this method, and each will be subject to uncertainties associated with its own test, thus the computed thermal conductivity will be subject to the combined uncertainties of all three components in the computation, and therefore be the least accurate.

15. Report

15.1 Conformance to this practice should be noted on the report described in Test Method **E1461** and the following statements should be added:

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

15.1.2 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;

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

15.1.4 Environmental surroundings of the specimen;

15.1.5 Statements of conformance with requirements of this standard.

15.2 Additionally, it is beneficial to report:

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

15.2.2 Energy pulse source;

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

15.2.4 Type of temperature rise detector.

15.2.5 Manufacturer and model of the instrument used.

16. Precision and Bias

16.1 A number of national and international round robin testing programs have shown that a measurement precision of $\pm 5\%$ can be attained for thermal diffusivity of a variety of materials. 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.

16.2 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.

16.3 Uncertainty analysis is to be performed for the instrument used for performing the measurements, and the results should be incorporated into the data analysis and reports.

17. Keywords

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

APPENDIX

X1. 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.

X1.1 For intrinsic thermocouples, the two legs of the thermocouple are not joined together in a bead, but are individually making contact with the specimen, thereby having the specimen 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.

X1.1.1 Intrinsic thermocouples can be used only with electrically conductive specimens or with non-conductive ones 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 specimen or this conductive layer.

X1.1.2 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.

X1.2 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 specimen.

X1.3 Intrinsic thermocouples are preferred over beaded couples.

X1.4 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.

X1.5 In the case of thermocouples, the response time (time to reach 95 % of steady-state value) can be defined (20) as follows:

$$t_{95} = \frac{25}{\pi} \cdot \frac{D^2}{\alpha_s} \cdot \frac{\lambda_T}{\lambda_s} \quad (\text{X1.1})$$

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. X1.1 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. X1.2:

$$\frac{T_i - T_0}{T_\infty - T_0} = 1 - (1 - a) \cdot e^{a^2 \cdot t^*} \cdot \text{Erfc}(a \cdot t^*) \quad (\text{X1.2})$$

where: T_0 and T_∞ are shown in Fig. X1.1, 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 constant wire on a copper substrate requires 3 μs to reach 95 % of steady-state. However, for the converse of this example, 25 μm copper wire on a constant substrate, it is found that 15 ms are required to reach 95 % of the steady-state. This is 5000 times slower than in the

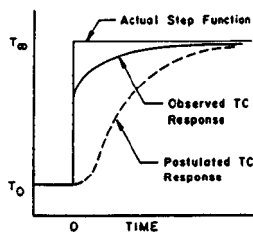


FIG. X1.1 Thermocouple Response Characteristics

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 X1.1 and Eq X1.2 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 X1.1).

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