



Designation: E1461-01 ~~Designation: E 1461 - 07~~

## Standard Test Method for Thermal Diffusivity by the Flash Method<sup>1</sup>

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 ( $\epsilon$ ) 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<sup>2</sup>/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.*, having applicability to much wider ranges of materials, applications, and temperatures, with improved accuracy of measurements.

1.3 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.

1.4 This test method is applicable to the measurements performed on essentially fully dense (preferably, but low porosity would be acceptable), homogeneous, and isotropic solid materials that are opaque to the specimen of applied energy pulse. Experience has shown, however, that some deviation from these strict guidelines can be accommodated with care and proper experimental design, substantially broadening the usefulness of the method.

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<sup>1</sup> 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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required. It is advisable to use reference materials to verify the performance of the instrument used.

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

1.7 For systems employing lasers as power sources, it is imperative that the safety requirement be fully met.

1.8 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:*<sup>2</sup>

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

E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples— Test Method for Thermal Diffusivity of Carbon and Graphite by Thermal Pulse Method

E 228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *thermal conductivity,  $\lambda$ , 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,  $\alpha$ , 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 *k*—constant in solution to diffusion equation. *C<sub>p</sub>*—specific heat capacity, J/(kg·K).

3.2.3 *L*—specimen thickness, meters. *k*—constant depending on percent rise.

3.2.4 *t*—response time, seconds. *K*—correction factors.

3.2.5 *K<sub>1</sub>, K<sub>2</sub>*—constants depending on  $\beta$ .

3.2.6 *L*—specimen thickness, m.

3.2.7 *t*—response time, s.

3.2.8 *t<sub>1/2</sub>*—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

3.2.10 *T*—temperature, Kelvin.

3.2.11 *T*—temperature, K.

3.2.11  $\alpha$ —thermal diffusivity, m<sup>2</sup>/s.

3.2.12  $\lambda$ —thermal conductivity, W/m·K.

3.2.13

3.2.12  $\beta$ —fraction of pulse duration required to reach maximum intensity.

3.2.14

3.2.13  $\rho$ —density, kg/m<sup>3</sup>.

3.2.14  $\lambda$ —thermal conductivity, W/m·K.

3.2.15  $\Delta T_5$ — $T(5t_{1/2})/T(t_{1/2})$ .

3.2.16

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 Test Method

4.1A small, thin disc specimen is subjected to a high-intensity short duration radiant energy pulse. The energy of the pulse is

<sup>2</sup> 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 information, refer to the standard's Document Summary page on the ASTM website.

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 (Summary of Test Method)

4.1 A small, thin disc specimen is subjected to a high-intensity short duration radiant energy pulse (Fig. 1). 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. 2). 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)<sup>3</sup> and review articles (3, 4, 5). A summary of the theory can be found in Appendix X5 Appendix X1.

**5. Significance and Use**

5.1 Thermal diffusivity is an important property, required for such purposes as design applications 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,  $\alpha$ , 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 Appendix X2).

5.4 Thermal diffusivity results, together with related values of specific heat capacity ( $C_p$ ) and density ( $\rho$ ) values, can be used in many cases to derive thermal conductivity ( $\lambda$ ), 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 samplespecimen and the radiative 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 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

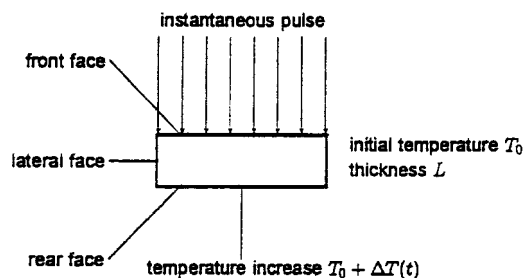
The essential components of the apparatus are shown in Fig. 3. These are the flash source, specimen holder, environmental enclosure (optional), temperature response detector and recording device.

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 (see 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).

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.03.

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



**FIG. 1 Schematic of the Flash Method**

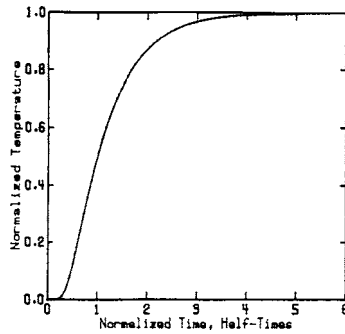


FIG. 2 Characteristic Thermogram for the Flash Method

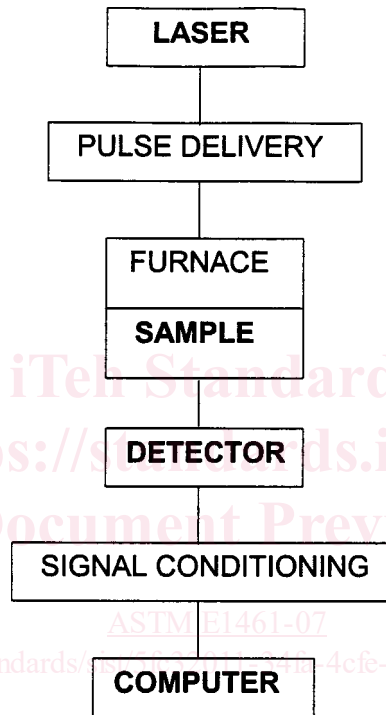


FIG. 3 Block Diagram of a Flash System

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), 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), *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 energy of the pulse hitting the specimen's surface must be spatially uniform in intensity.

7.2 An environmental control chamber is required for measurements above and below room temperature.

7.3 The detector can be a thermocouple, infrared detector, optical pyrometer, or any other sensor 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 not more than 2 % of the half-rise time value.

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

#### 7.5 *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 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 later sections.)

7.6.4 In case the recording device does not have accurate built-in timing (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.

7.5.1 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 Measurement of specimen's 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 specimen. Touching the specimen with thermocouples is



not recommended. Embedding thermocouples into the sample is not acceptable.

7.7 The temperature controller and/or programmer are to bring the specimen to the temperatures of interest.

## 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.

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 (in special cases, as small as 6 mm diameter and as large as 30 mm diameter have been reported as used successfully). 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.

8.2 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 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

8.3 *Specimen Surface Preparation* —It is a good practice to apply a very thin, uniform graphite or other high emissivity coating on both faces of the specimen 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 specimen to absorb the energy applied, especially in case of highly reflective materials. For transparent materials, a layer of gold, silver, or other opaque materials must be deposited first, followed by graphite coating. For some opaque reflective materials, grit blasting of the surface can provide sufficient pulse absorption and emissivity, especially at higher temperatures, where coatings may not be stable or may react with the material.

## 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 periodically 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-rise 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 (appropriate heat loss corrections being applied) 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.