Designation: E 1461 – 07

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 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, 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), homogeneeous, 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.

1.5 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.

1.6 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:* [2]

C 714 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, 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 $t_{½}$ —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, m^2/s.

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

3.2.13 ρ—density, kg/m^3.

3.2.14 λ—thermal conductivity, W/m·K.

3.2.15 Δt_5—$T\,(5t_{½})\,/T\,(t_{½})$.

3.2.16 Δt_{10}—$T\,(10t_{½})\,/T\,(t_{½})$.

[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] 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.

3.2.17 ΔT_{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

4.1 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**)[3] and review articles (**3, 4, 5**). A summary of the theory can be found in Appendix X1.

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 Appendix X2).

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:

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

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

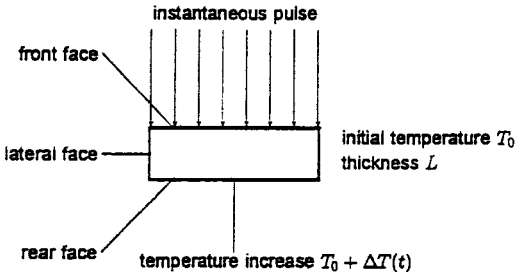

FIG. 1 Schematic of the Flash Method

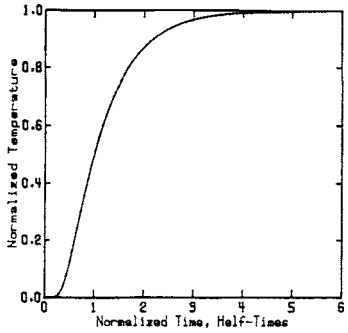

FIG. 2 Characteristic Thermogram for the Flash Method

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 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 in performing a rigorous evaluation of these factors are given in (**31**).

7. Apparatus

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), 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.

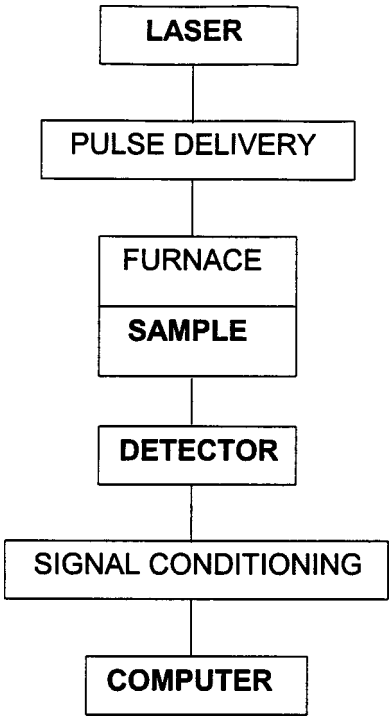

FIG. 3 Block Diagram of a Flash System

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*

7.5.1 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 10 to 12.5 mm in diamete (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 iether 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 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

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

10.2 The testing procedure must contain the following functions:

10.2.1 Determine and record the specimen thickness.