



Standard Test Method for Thermal Diffusivity of Solids by the Flash Method¹

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 homogeneous solid materials. Thermal diffusivity values ranging from 0.1 to 1000 mm²/s are readily measurable by this test method and measurements can be made from about 100 to 2500 K normally in a vacuum or inert gas environment.

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 measurement.

1.3 This test method is applicable to the measurement of a wide variety of homogeneous opaque materials and, with special precautions, can be used on transparent and some porous and composite materials.

1.4 This test method is intended to allow a wide variety of apparatus design and design accuracies to satisfy the requirements of specific measurements problems. 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 can be considered an absolute (or primary) method of measurement since no heat flux reference standards are required except for verification purposes and to confirm accuracy statements.

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

1.7 *This standard does not purport to address all of the safety problems, 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 Method²

E 220 Method for Calibration of Thermocouples by Com-

parison Techniques^{2, 3}

E 230 Temperature-Electromotive Force (EMF) Tables for Thermocouples³

3. Terminology

3.1 Definitions:

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.

3.1.1.1 *Discussion*—Where other modes of heat transfer are present in addition to conduction, this property is often referred to as apparent or effective thermal conductivity, e or *app*.

3.1.1.2 *Discussion*—For practical purposes, the lateral extent of a slab is considered to be infinite when heat flow laterally is less than 2 % of the transverse flow.

3.1.1.3 *Discussion*—The property must be identified with both a specific mean temperature, since it varies with temperature, and for a direction and orientation of thermal transmission since some bodies are not isotropic with respect to the thermal conductivity.

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 Definitions:

3.2.1 a — $1/(1 + 0.667 \lambda_T/\lambda_s)$.

3.2.2 D —diameter, meters.

3.2.3 k —constants in solution to diffusion equation.

3.2.4 L —specimen thickness, meters.

3.2.5 t —response time, seconds.

3.2.6 t^* —dimensionless time ($t^* = 4\alpha_s t/D^2T$).

3.2.7 T —temperature, Kelvins.

3.2.8 α —thermal diffusivity, m²/s.

3.2.9 λ —thermal conductivity, W/m.K.

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

3.2.11 Δt_5 — $T(5t^{1/2}/T(t^{1/2}))$.

3.2.12 Δt_{10} — $T(10t^{1/2}/T(t^{1/2}))$.

3.3 Definitions:

3.3.1 o —ambient.

3.3.2 s —specimen.

3.3.3 T —thermocouple.

3.3.4 x —percent rise.

¹ This test method is under the jurisdiction of ASTM Committee E-37 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 15.01.

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

- 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 mounted horizontally or vertically is subjected to a high-intensity short duration thermal pulse. The energy of the pulse is absorbed on the front surface of a specimen and the resulting rear face temperature rise is measured. The ambient temperature of the specimen is controlled by a furnace or cryostat. Thermal diffusivity values are calculated from the specimen thickness and the time required for the rear face temperature rise to reach certain percentages of its maximum value. This test method is described in detail in a number of publications (1, 2)⁴ and review articles (3, 4, 5) and has been standardized in Test Method C 714 in a more simple form for carbons and graphites.

NOTE 1—While this test method was developed for and applied originally to homogeneous opaque solids, in which a large front face temperature excursion was not detrimental, 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).

4.2 The pulse raises the temperature of the specimen only a few degrees above its initial ambient value. However, initially all of this energy is deposited on the front surface and this temperature may rise many degrees. If this should damage the specimen, a layer of a material with known properties (thermal diffusivity, specific heat and density) and measured thickness can be attached to the front surface and results on the composite specimen analyzed using a two-layer method. This requires that the specific heat and density of the specimen also be known (13).

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 the simple specimen geometry, small specimen size requirements, rapidity of measurement, and ease of handling materials having a wide range of thermal diffusivity values over a large temperature range with a single apparatus. The short measurement times involved reduce the chances of contamination and change of specimen properties due to exposure to high temperature environments.

5.3 Thermal diffusivity results in many cases can be combined with values for specific heat (C_p) and density (ρ) and used to derive thermal conductivity (λ) from the relation $\lambda = \alpha C_p \rho$.

6. Interferences

6.1 Experimental results are subject to two general types of errors:

6.1.1 Measurement errors associated with uncertainties that exist in measured quantities used to compute the thermal diffusivity from experimental data. The units of thermal diffusivity are length and time. Because test specimens are relatively thin (generally 1.5 to 4 mm) and the thickness enters as a squared term, uncertainties in the determination of the thickness can be very significant. The time is that observed for the rear face temperature to attain a certain percentage of the maximum rise. This latter measurement involves determining a base line, the maximum rise above the base line, and the time of initiation of the heat pulse as an integral part of the time determination. Response times of the detectors and amplifiers are extremely important. Response time effects are considered in detail in 7.1.3. Measurement errors have been discussed in some detail (5, 14, 15).

6.1.2 Non-measurement errors associated with deviations of actual experimental conditions as they exist during the experiment from the boundary conditions assumed in the mathematical model used to derive the equation for computing the thermal diffusivity. The major sources of non-measurement errors are finite pulse time effect, heat losses or gains, and non-uniform heating. The mathematical derivation assumes that the energy pulse is delivered in times short compared to the rise time. The case where this is not true is called the finite pulse time effect and it becomes important when the duration of the energy pulse is greater than 2 % of the half rise time. It should be noted that these three effects are not strictly classified as errors (may be determined experimentally) but are merely deviations from an ideal situation in which these effects are assumed to be negligible. It is assumed that the material is opaque, that is, radiation from the energy source heats only the specimen surface. Furthermore, it is assumed that the temperature sensor follows accurately the rear surface temperature excursion. Thus, if an i.r. detector is used, it must not view into the specimen interior. Specimens of translucent/transparent materials require special techniques similar to those used for layered structures.

7. Apparatus

7.1 The essential features of the apparatus are shown in Fig. 1. These are the flash source, sample holder and environmental

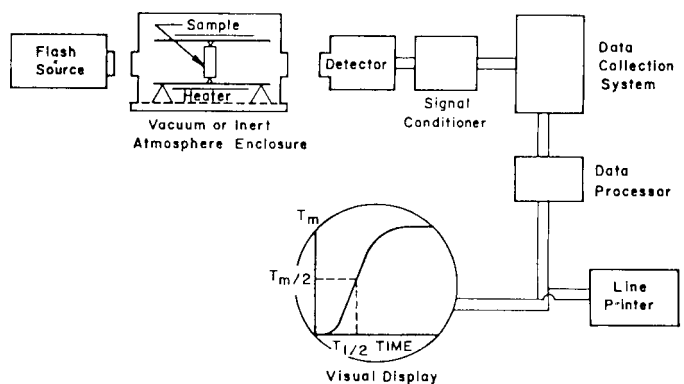


FIG. 1 Flash Diffusivity Apparatus (Schematic)

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

system, temperature response detector, and data collection and analysis components.

7.1.1 The flash source may be a laser, a flash lamp, or an electron beam. The duration of the energy flash should be less than 0.02 of the time required for the rear face temperature rise to reach one-half of its maximum values (see Fig. 1). If this condition is not met, it is necessary to correct the data for the finite pulse time effect (16, 17, 18, 19). The energy source shall have uniform intensity over the front surface of the specimen. The rear face temperature rise shall be kept to a few Kelvin.

7.1.2 An environmental control chamber for vacuum or inert gas environment is required for measurements above and below room temperature. Unless the source is enclosed within the chamber, for example, as for the case of an electron beam source, the enclosure shall be fitted with a window which is transparent to the flash source. A second window is required if optical detection of the temperature rise curve is used and the optical detector must be shielded from direct exposure to the energy beam.

7.1.2.1 The furnace or cryostat should be loosely coupled (thermally) to the specimen support system and shall be capable of maintaining the specimen temperature constant within 4 % of the maximum temperature rise over a time period equal to five half rise times. The furnace may be horizontal or vertical. The specimen support shall also be loosely coupled thermally to the specimen point contacts or equivalent supports. Depending on specimen orientation point contacts or equivalent supports, constructed with low thermal diffusivity materials, are the preferred means.

7.1.3 The detector can be a calibrated thermocouple (see Method E 220 and Tables E 230), infrared detector or automatic optical pyrometer. It shall be capable of detecting 50 mK change above the ambient temperature. It is desirable that the detector response be linear with temperature over a few degrees and that the rear face temperature rise be limited to this range. The time response of the detector and its associated amplifier is extremely important. While the response time of the detectors in optical instruments is often orders of magnitude faster than required for the flash method, the detector signal is fed into amplifiers and filters having response times which can be slow enough to effect transient readings. Therefore, the response time of the total circuit must be checked using choppers or other devices to prove that it is less than 0.1 of the half time value. The use of electronic filters is discouraged as they tend to distort the shape of the time-temperature curve.

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

$$t_{0.95} = \frac{25}{\pi} \frac{D_T^2 \lambda T}{\alpha_s \lambda_s} \quad (1)$$

Thus, a small diameter thermocouple of low conductivity material attached to a specimen of a high conductivity and high diffusivity material yields the fastest response time. Eq 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 2:

$$\frac{T_t - T_0}{T_\infty - T_0} = 1 - (1 - a)e^{-a^2 t^*} \operatorname{Erfc}(a t^*) \quad (2)$$

where:

T_0 and T_∞ are shown in Fig. 2, t^* is dimensionless time ($t^* = 4\alpha_s t/D^2 T$), 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 μg 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 fold slower than 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 intrinsic thermocouples (21).

7.1.3.2 Eq 1 and Eq 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 spotweld 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 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 diffusivity to measure specimens of materials of low thermal diffusivity, that can lead to very large response times (see Eq 1).

7.1.3.3 When using remote temperature sensing, several precautions are required. The sensor must be focused on the center of the back surface. The sensor 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 and proper shielding is necessary. For protection against lasers, dielectric spike filters, that are opaque at the selected wavelength, are

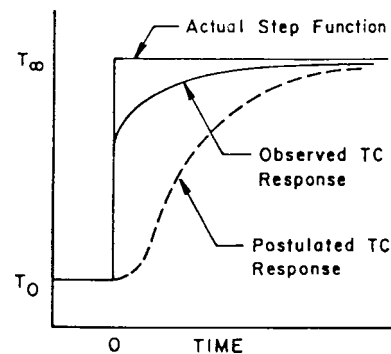


FIG. 2 Thermocouple Response Characteristics

very useful. The viewing window and any focusing lenses must not absorb appreciably in the wavelength region of the sensitive 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 any deposit build-up with resulting absorption of energy. Such build-up can lead to loss of sensitivity of the optical detector and reduced or non-uniform specimen heating from the pulsed energy source.

7.1.4 The signal conditioner includes the electronic circuitry to bias out the ambient temperature reading, spike protectors, amplifiers and analog-to-digital converters. Precautions shall be taken to prevent driving the components into saturation and to have adequate response time so that the transient signal is not distorted.

7.1.5 The data collection system may be a digital data acquisition system, digital oscilloscope or a regular oscilloscope. In the latter case, provisions must be made to photograph the trace. The timing measurements are provided by the data collection system and the accuracy of the time base shall be verified so that half rise time readings are measured within 0.2%. The starting time for the time duration shall be based on the initiation of the actual energy pulse and not on the time the laser power supply started to charge or the time the flash lamp started to discharge.

7.1.6 The data processor may be a simple adjustable scale for measuring the elapsed time on a photograph plus a hand-held calculator, a digital oscilloscope, or a digital data acquisition system. It is important that the transient response curve be analyzed to verify the presence or absence of finite pulse time effects, radiation heat losses and non-uniform heating.

7.1.6.1 As a minimum, this means computing the thermal diffusivity using the 25, 50, and 75 % rise times and ideally this means comparing the normalized rise curve with the theoretical model.

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 diffusivity and should be chosen so that the half time 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 1.5 to 4 mm range. Since the half time 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 Measure the specimen thickness and maintain its uniformity to an accuracy of 0.5 % of the thickness.

9. Calibration and Verification

9.1 Calibrate both the micrometer used to measure the specimen thickness and the time base of the data acquisition so that the thickness and time measurements are accurate to

within 0.2 %. However, it must be recognized that the most serious and insidious errors arise from inadequate time response of the detector and its associated electronics and from nonobservance of the boundary conditions assumed in the mathematical mode, that is, non-uniform heating, heat losses, or finite pulse-time effects. The response time can be checked by the measurements on a standard specimen or reference material having a thickness estimated to cause about the same half time as the unknown. The presence of the other effects is detected by the methods given in Section 11.

9.2 While there are no standard reference materials (SRM) issued by National Institute of Standards and Technology (NIST) specifically for thermal diffusivity, several materials can be used as reference materials. POCO graphite is available from NIST and thermal diffusivity values for it and several other materials have been established through round-robins (22, 23). Armco iron and cordierite are other materials that have been used as reference materials. 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).

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 diffusivity values) are closely similar to that of the unknown and the temperature-rise curves are determined in an identical manner for the reference and unknown. One important check of the validity of data (in addition to the comparison of the rise curve with the theoretical model) 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 diffusivity value with representative specimens of significantly different thicknesses or better yet, on the same sample with different specimen thicknesses, then the results can be assumed valid.

10. Procedure

10.1 Determine and record the specimen thickness.

10.2 Mount the specimen in its holder. Attach thermocouples where appropriate.

10.3 Align the specimen in the path of the energy beam. If the source is a laser, a helium-neon alignment laser aimed through the solid state laser is a convenient method of determining the energy beam path.

10.4 Aperature the energy beam path so that only the front surface of the specimen will be subjected to the energy pulse. This minimizes stray radiation that can affect the detector and cause base-line offsets.

10.5 Where an infrared detector is used to monitor the temperature rise, align it centrally on the rear surface.

10.6 Establish a vacuum or inert gas environment in the chamber.

10.7 Energize the power source, taking proper safety precautions. Pulse the source and observe the rear face temperature rise. Adjust the amplification, biasing, and spike protection circuits as necessary to obtain a readily detectable temperature rise curve.