



Standard Test Method for Thermal Diffusivity of Carbon and Graphite by a Thermal Pulse Method¹

This standard is issued under the fixed designation C 714; 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 carbons and graphite to $\pm 5\%$ at temperatures up to 500°C. It requires only a small easily fabricated specimen. Thermal diffusivity values in the range from 0.04 to 2.0 cm²/s are readily measurable by this test method; however, for the reason outlined in Section 5, for materials outside this range this test method may require modification.

1.2 *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. Summary of Test Method

2.1 A high-intensity short-duration thermal pulse from a flash lamp is absorbed on the front surface of a specimen; and the rear surface temperature change as a function of time is observed on an oscilloscope. The pulse raises the average temperature of the specimen only a few degrees above its initial value. The ambient temperature of the specimen is controlled by a furnace or cryostat. Thermal diffusivity is calculated from the specimen thickness and the time required for the temperature of the back surface to rise to one half of its maximum value (**1**).²

2.2 The critical factors in this test method are:

2.2.1 $\tau/t_{1/2}$ must be 0.02 or less. τ is the pulse time as defined in Fig. 1 and $t_{1/2}$ is the time for the rear surface temperature to rise to one half of its maximum value (see Fig. 2).

2.2.2 Heat losses from the specimen via radiation, convection, or conduction to the specimen holder must be small. Whether or not this condition is violated can be determined experimentally from the oscilloscope trace, an example of which is shown in Fig. 2. If $\Delta T(10 t_{1/2})/\Delta T(t_{1/2}) > 1.98$, the heat losses are assumed to be zero.

2.2.3 The oscilloscope trace must be such that ΔT_{\max} , $\Delta T(10 t_{1/2})$, and $t_{1/2}$ can be determined to $\pm 2\%$.

2.2.4 The other conditions are less critical, and the experimenter is left to his discretion.

3. Significance and Use

3.1 Sulfur, even in very low concentrations, is of concern in a nuclear reactor because of potential corrosion of metallic components. This test method has the sensitivity to analyze very low sulfur contents in graphite using very small samples.

3.2 This test method can be used to characterize graphite for design purposes.

4. Apparatus

4.1 The essential features of the apparatus are shown in Fig. 3. The window may be any material that is transparent to the flash source. The specimen holder should be a ceramic or other material whose thermal conductivity is low relative to that of the sample.

4.2 *Thermocouple*, used to monitor the transient temperature response of the rear surface of the specimen. The wire ends should be prepared to minimize heat losses from the specimen to the thermocouple wires (that is, by grinding to points or clipping) and attached in a manner that prevents penetration into the specimen. They are separated by about 1 mm so that the electrical circuit of the thermocouple is completed through the specimen.

4.3 *Oscilloscope*, with calibrated sweep speeds that can be varied from 0.1 ms/cm to 0.5 s/cm or more. The vertical amplifier section of the oscilloscope should have a frequency response in the range from 0.06 to 10 kHz to be perfectly insensitive to frequency in the range of interest described in Section 5. A minimum vertical deflection sensitivity of 1 C/cm is recommended. The cathode-ray tube should have a usable viewing area of at least 40 by 100 mm. A camera is used to photograph the oscilloscope trace.

4.4 *Flash Tube*—The experimenter has considerable latitude in his choice of flash tube. A typical 1000-J unit raises the specimen temperature from 1 to 3°C. The power supply for such a unit might consist of a 125- μ F capacitor bank charged to 4000 V; discharge time would be about 1 ms. Either an external

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² The boldface numbers in parentheses refer to the list of references at the end of this test method.

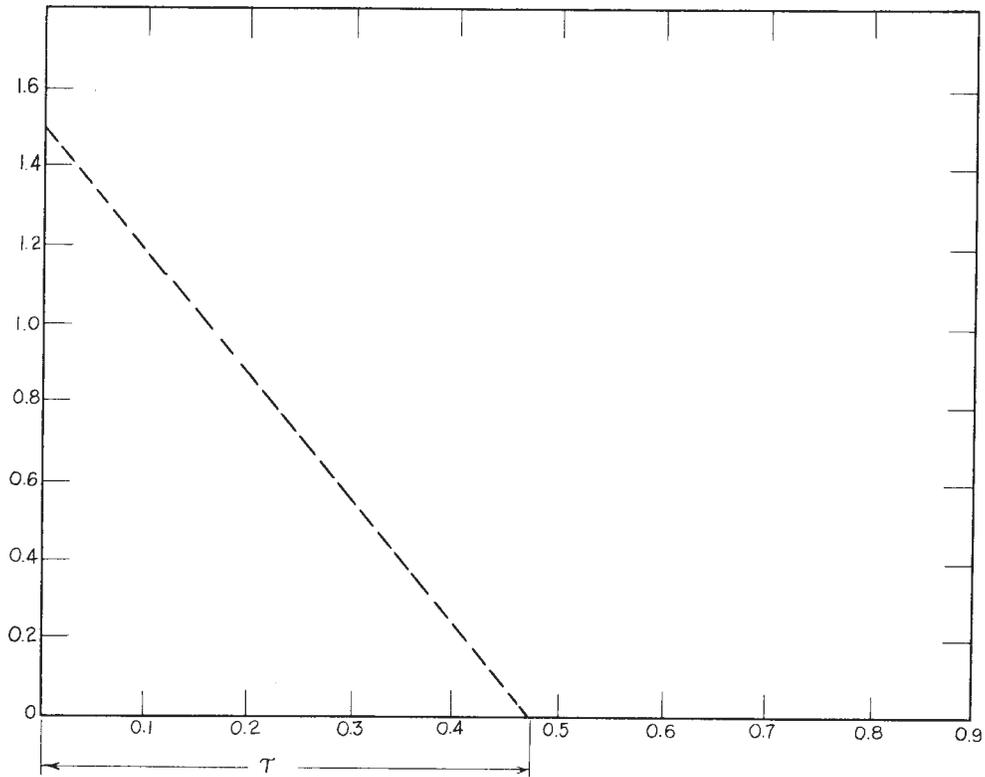


FIG. 1 Flash Tube Response

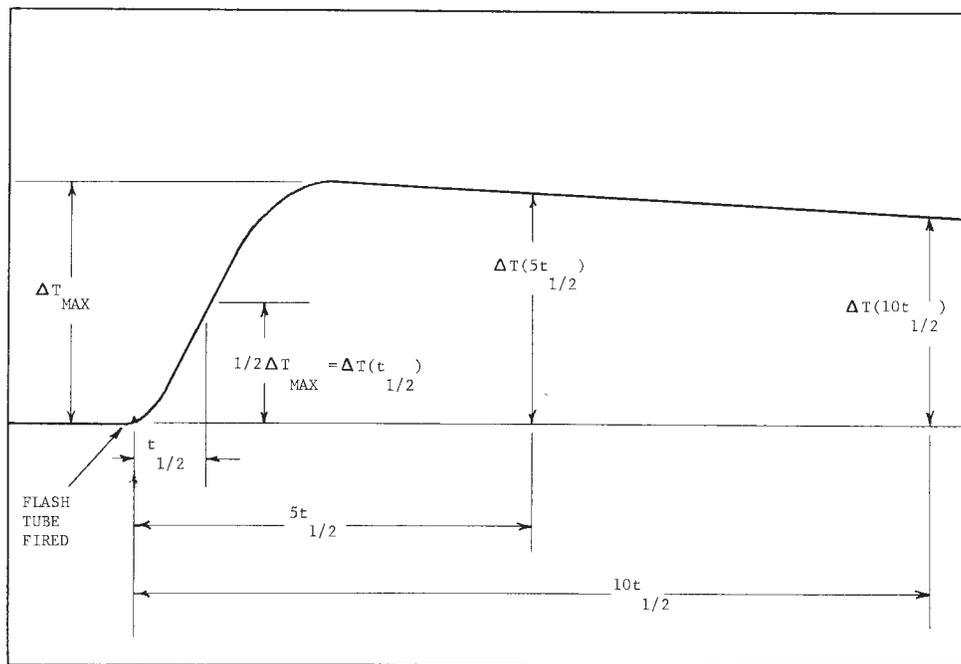


FIG. 2 Example of Oscilloscope Trace Showing Parameters Used to Calculate Thermal Diffusivity

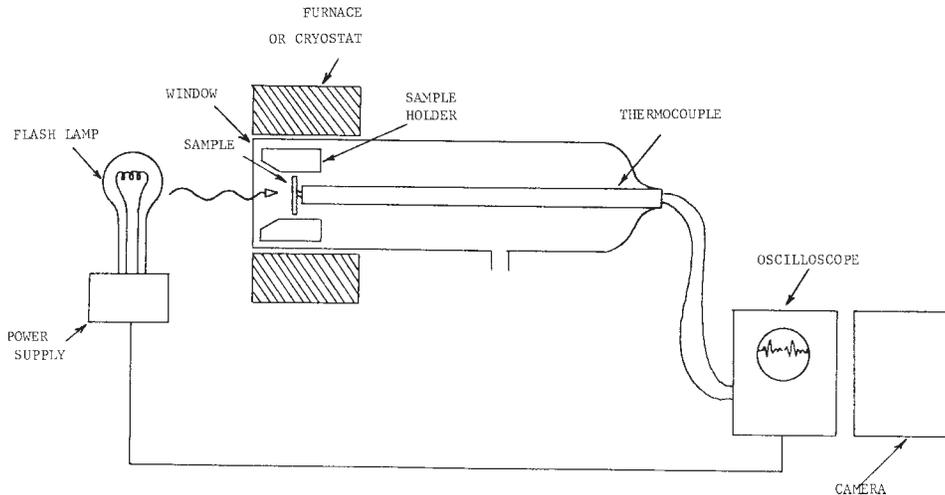


FIG. 3 Schematic Diagram of Apparatus

trigger device or a delayed trigger pulse from the oscilloscope may be used to fire the flash tube.

5. Test Specimen

5.1 The specimen shall be a circular disk, 2 to 4 mm thick and 6 to 12 mm in diameter; however, several things must be considered in choosing specimen dimensions. The diameter is fairly arbitrary except that it must not be too large relative to the flash source because the front surface of the specimen must be illuminated uniformly and, therefore, heated uniformly. Specimen thickness must be selected so that $\tau/t_{1/2} < 0.02$, where τ is the pulse time, and $t_{1/2}$ is defined as in Section 2 and by Fig. 2. However, the temperature-rise time must not be so long that heat is also lost radially to the specimen holder. In meeting these criteria, the time for the rear surface temperature to reach one half its maximum should be between 0.02 and 0.10 s.

5.2 The specimen thickness should be measured with an accuracy of ± 0.01 mm. Front and rear surfaces should be parallel to within ± 0.01 mm and the surfaces should be flat to within ± 0.01 mm.

6. Calibration

6.1 Since this is an absolute method, no calibration *per se* is required. However, the accuracy of the equipment should be certified by measuring the thermal diffusivity of a suitable standard in the temperature range of interest, for example, Armco iron.

6.2 The oscilloscope sweep rate shall be calibrated with a time mark generator.

7. Procedure

7.1 Mount the specimen in its holder and place the thermocouple in contact with the rear surface of the specimen. Position the specimen holder inside the specimen chamber, and place the assembly in the furnace or cryostat. An inert gas or

vacuum may be required for measurements above about 300°C. The atmosphere in the specimen chamber shall be such that specimen mass loss is held to less than 0.5 %. Energize the power supply for the flash tube and generate a thermal pulse. Observe the temperature change on the oscilloscope and make adjustments to the sweep rate, if necessary, before pulsing again for a photograph of the trace.

8. Calculation

8.1 Calculate the thermal diffusivity, α , as follows:

$$\alpha = \omega L^2 / t_{1/2}^2$$

where:

L = thickness of the specimen, cm,

$t_{1/2}$ = time for the rear surface temperature to rise to one half of its maximum value, s, and

ω = parameter that is a function of the heat loss.

For the ideal case of zero heat loss [$\Delta T(10 t_{1/2}) / \Delta T(t_{1/2}) > 1.98$] and sufficiently small pulse width ($\tau/t_{1/2} < 0.02$), $\omega \cong 0.139$.

8.2 Where heat losses from the sample are significant or where the duration of the thermal pulse is not sufficiently short, techniques have been developed for applying the necessary corrections (2,3,4,5).

9. Report

9.1 The report shall include the following:

9.1.1 Thermal pulse source,

9.1.2 Method of calculation,

9.1.3 Identification and previous history of the test specimen,

9.1.4 Ambient temperature of the specimen,

9.1.5 Calculated value of thermal diffusivity, and

9.1.6 Density of the specimen.

REFERENCES

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