Standard Test Method for Determination of Thermal Conductivity of a Graphite Foil

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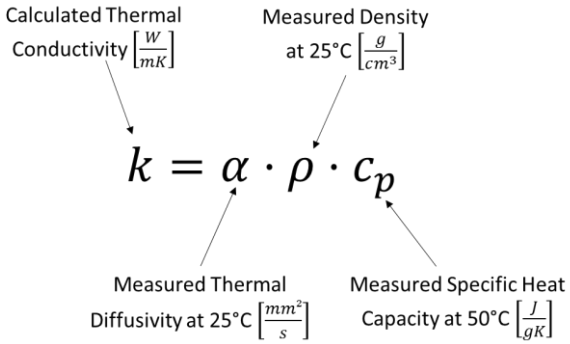
1.0 Introduction

1.1 **Purpose** The purpose of this document is to establish a standard method to calculate the thermal conductivity of a graphite foil from the measured bulk density, specific heat capacity, and thermal diffusivity of a specimen. The document will provide a procedure to measure each property in the calculation.

1.2 **Scope** This standard covers a test method to determine the average in-plane thermal conductivity of a graphite foil having a thickness of 0.010mm to 0.150mm. The specimen should be a single homogenous layer of bare graphite (no coatings, plastics, adhesives, etc.) This standard applies only to the determination of thermal conductivity in the specimen test range of 20°C to 50°C since thermal conductivity is a function of temperature. All values are to be stated in SI units.

2.0 Thermal Conductivity Determination



2.1 Equation Definitions

2.1.1 k denotes the calculated specimen in-plane thermal conductivity in SI units Watts per meter per degrees Kelvin, $\frac{W}{mK}$. The value is based on the empirical measurement of three intrinsic material properties; thermal diffusivity, density, and specific heat capacity.

2.1.2 α denotes the measured specimen thermal diffusivity in SI units, square millimeters per second, $\frac{mm^2}{s}$. This measurement should be taken at an environment and sample temperature of 25°C.

2.1.3 ρ denotes the measured specimen density in SI units, grams per cubic centimeter, $\frac{g}{cm^3}$. This measurement should be taken at an environment and sample temperature of 25°C.

2.1.4 c_p denotes the measured specimen specific heat capacity at constant pressure in SI units Joules per gram per degrees Kelvin, $\frac{J}{gK}$. This measurement should be taken at an environment and sample temperature of 50°C.

3.0 Measurement Methodology

3.1 Thermal Diffusivity

3.1.1 Thermal diffusivity should be tested on a piece of equipment specifically designed to test anisotropic, thin, high thermal conductivity materials. Equipment should be properly maintained; calibrated with weekly checks against a test standard and monthly calibration.

3.1.1.1 Ideal test methods are those that irradiate one surface of the specimen with a laser heat pulse while monitoring the heat diffusion on the opposite side of the sample at varying distances from the laser source. Acceptable test methods include but are not limited to the Angstrom method, Distance-Variation method, radial heat flow method, and other methods using AC periodic laser heating.

3.1.1.1.1 Examples of acceptable thermal diffusivity measurement equipment include but are not limited to:

- Bethel TA33 Thermowave Analyzer
- LonGwin LW-9614 In-Plane Thermal Diffusivity and Thermal Conductivity Measurement Apparatus
- Netzsch LFA 467 HyperFlash®
- ULVAC LaserPIT AC Method Thermal Diffusivity Measuring

3.1.1.2 Unacceptable test methods include Guarded Hot Plate method (GHP), ASTM D5470 method, Heat Flow Meter method (HFM), 3ω-method, 2Ω-method, Time-Domain Thermoreflectnace (TDTR) method, and Hot Disk Analysis method.

3.1.2 The test equipment should be able to handle sample thicknesses ranging from at least 10 to 200µm. Instrument repeatability should be less than ±5%. Ideally, the heat diffusion should be measured with a non-contact sensor such as IR (infrared radiation).

3.1.3 The test specimen should be flat, bare graphite without cosmetic anomalies or physical damage (rips, rough edges, folds, wrinkles, etc.) Ideally, sample size is at least 25cm² in area. The sample should be a single layer of graphite without coatings, laminates, plastics, or adhesives.

3.2 Density

3.2.1 The volumetric mass density of the specimen is calculated by dividing the mass of the specimen by its volume. Ideally, the test specimen is the same sample that was used to measure the thermal diffusivity.

3.2.1.1 The density tests specimen should be no smaller than 25cm²

3.2.1.2 Specimen mass should be measured using a properly sized and calibrated laboratory scale with no less than 0.001g resolution. The specimen should be shielded from surrounding (i.e. air movement) during measurement.

3.2.1.3 Specimen volume can be calculated using two methods, a physical measurement (length x width x thickness) or using gas pycnometry (gas displacement method).

3.2.1.3.1 Physical Measurement Method

- Specimen length and width should be maintained using a die to minimize measurement error, sample preparation damage, and to minimize sample-to-sample variation.
- Specimen thickness should be determined using a properly calibrated micrometer or thickness gauge with a resolution of 0.001mm or better and an accuracy of no more than ±0.002mm. The

measurement anvil diameters should be between ø4mm and ø20mm. The thickness used to calculate density should be an average of at least 5 measurements evenly spaced on the specimen.

3.2.1.3.2 Gas Pycnometry Measurement Method

- A Gas Pycnometer uses a measurement of gas displacement to determine the volume of a test specimen. Typically helium or nitrogen are used as the working gas.
- A gas pycnometer should have a minimum 1 cc test chamber and a specimen should be chosen such that is fills at least 60% of the test chamber volume (≥0.6 cc).

3.2.1.4 Density will be calculated by dividing the specimen mass by the specimen volume in $\frac{g}{cm^3}$.

3.3 Specific Heat Capacity

3.3.1 Specific Heat Capacity should be measured using a differential scanning calorimeter (DSC) or test equipment including DSC functionality. Utilizing a Temperature-Modulated Differential Scanning Calorimetry (TM-DSC) method is preferred.

3.3.1.1 Examples of acceptable specific heat capacity measurement equipment include but are not limited to:

- Linseis DSC PT1000
- Netzsch DSC 214 Polyma or DSC3500 Sirius
- TA Instruments Discovery 25/250/2500 DSC
- TA Instruments Q20/Q200/Q2000 DSC

3.3.1.2 Sample preparation is critical to yield accurate specific heat capacity results. Air or contamination in the sample pill will add to variation and error in the measurement values as will poor placement of the test pill into the DSC tester.

3.3.1.3 The preferred method of sample preparation utilizes a die to cut out small circles of the graphite foil that will fit tightly in the sample pill. Ideally, sample material will be from the area surrounding the thermal diffusivity and density test specimen(s). Layers of the graphite foil circles are stacked tightly in the pill until they weigh between 10 and 30mg (target 20mg) and then the pill can be sealed. It is critical that the graphite foil circles are flat and properly sized to minimize the air trapped in the pill.

3.3.1.4 Using the TM-DSC method, an example test method is as follows:

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1: Modulate +/- 2.00°C every 120 seconds
2: Data storage: Off
3: Equilibrate at 50.00°C
4: Data storage: On
5: Isothermal for 10.00 min
6: End of method
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3.3.1.5 If temperature modulation is unavailable, a standard DSC method can be used however, it is critical to begin testing at -40°C or colder to ensure that a linear heating rate can be established before approaching the 50°C test temperature. This will require the testing unit to have a chiller option installed. A heating rate of no more than 20°C per minute should be used for testing.

3.3.2 The value of specific heat for a given graphite foil product can be taken as a constant for purposes of process control and shipment certifications. This can only be done if the raw material and all processing parameters/equipment remain unchanged after a product has been developed and qualified. Sufficient data must been collected demonstrating process capability (CPK ≥1.33) with a tolerance of $\pm 0.10 \frac{J}{gK}$. Additional periodic checks should be completed (i.e. once per run/day/lot) and documented in a control chart to ensure a stable average is maintained.

4.0 Revision Summary

Revision Number:	Date (YYYYMMDD):	Editor(s):	Description of Change:
01	20160630	JAT	Initial Release
02	20160718	JAT	Updated List of Acceptable Equipment
03	20161202	JAT	Added Gas Pycnometry Volume Measurement to Density Calculation Method
04	20181024	JAT	Updated to NeoGraf