National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Report

NMIJ RM 1401-a
No. +++

Thermal Conductivity Reference Material (Isotropic Graphite)

This reference material (RM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This RM is intended for use in calibration of thermal conductivity analyzers. It is also intended to be used for validation of analytical techniques and instruments.

Indicative Values

The indicative values (thermal conductivity λ) of this RM at typical temperatures and its uncertainties are given in the table below. The uncertainty of each indicative value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Temperature	Thermal conductivity	
T (K)	λ (W m^{-1} K^{-1})	U_λ (%)
300	126	9.8
400	110	8.4
500	99.4	7.7
600	90.5	7.4
700	83.1	7.5
800	76.5	7.6
900	70.6	7.7

The thermal conductivity λ (Wm^{-1}K^{-1}) in the table is calculated by using the following equation:

$$\lambda(T) = \alpha(T) \times \left(c_p(T) \times 1000\right) \times \rho$$

See the Reference Information given in this report for the thermal diffusivity α (m^{2}s^{-1}), the specific heat capacity c_p (J K^{-1} g^{-1}) and the bulk density ρ(kg m^{-3}) in the following page.

Analysis

The indicative value (thermal conductivity λ) of this RM is calculated as a product of the thermal diffusivity α, the specific heat capacity c_p, and the bulk density ρ. The values of thermal diffusivity α, specific heat capacity c_p and bulk density ρ are obtained using the methods described below. (See【Technical Information】for their values.)

The thermal diffusivity α was measured by the laser flash method in accordance with the procedures for the NMIJ's calibration service on thermal diffusivity (QMC TD). Five samples for the thermal diffusivity evaluation (with a diameter of 10 mm and with a thickness of 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm and 4.0 mm) were prepared from each of ten isotropic graphite rods (with a diameter of 10 mm and with a length of 100 mm) for distribution, which were examined to determine the value of thermal diffusivity. To be more specific, the homogeneity was first evaluated based on the room-temperature measurement results for all the samples, and then by using all the sample of 2.0 mm in thickness and a pair composed of samples with different thicknesses, thermal diffusivity was measured at room temperature, about 500 K, 700 K and 900 K. Based on the all measurements, the equation of temperature dependence was obtained through the least squares method.

The specific heat capacity c_p was measured by the Differential Scanning Calorimetry (the DSC method) in accordance with the procedures for commercial test of specific heat capacity (QMC SH). A sample for the specific heat capacity evaluation (with a diameter of 5 mm and with a thickness of 1.0 mm) was prepared from each of ten rods for distribution, which was measured at about 360 K, 550 K, 740 K and 930 K to determine the value of specific heat capacity. Based on the all measurements, the equation of temperature dependence was obtained through the least squares method.

The bulk density ρ was measured at room temperature by using the method stipulated in JIS R 7222. Samples for the bulk density evaluation (with a diameter of 10 mm and with a thickness of 10 mm) were prepared from each of ten rods for distribution, which were evaluated to determine the value of bulk density.

The tests to assign values were performed under the following conditions:

Range of temperatures: 300 K to 900 K

Ambience: the thermal diffusivity: in the air ambience (at room temperature) and in vacuum (at less than 1.0 Pa) (higher than room temperature),
the specific heat capacity: in the nitrogen gas ambience

Metrological Traceability

The thermal diffusivity α of this RM is determined by using the thermal diffusivity measurement system calibrated in a way to be traceable to the International System of Units (SI). The specific heat capacity c_p, which is also the reference values of this RM, is determined by using the equipment traceable to the SI and the reference material. The bulk density ρ, which is the third reference values of this RM, is determined by using the equipment traceable to the SI. Therefore the thermal conductivity λ, the indicative value of this RM, which is obtained as a product of these three, is traceable to the SI.

Expiration of Report

This report is valid from the date of shipment to March 31, 2025, provided that the material is stored in accordance with the instructions given in this report.

Sample Form

This RM consists of a pair of its black disc-shaped samples packaged in a plastic container: one with a diameter of 10 mm and a thickness of 1.0 mm and the other with a diameter of 10 mm and a thickness of 2.0 mm.

Homogeneity

Variation of the indicative values derived from the inhomogeneity was evaluated as variation of the thermal diffusivity which is one of the reference values. Five samples for the thermal diffusivity evaluation (with a diameter of 10 mm and with a thickness of 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm and 4.0 mm) were prepared from each of ten rods for distribution, and the thermal diffusivity of all these samples was measured at room temperature. This RM is considered homogeneous within U since the homogeneity is reflected in the uncertainty of the indicative value λ.

Instructions for Storage

This RM should be stored at a temperature of 23 °C ± 5 °C and at a relative humidity of 50 % or less.

Instructions for Use

The information on the use of this RM is given below:

- This RM must not be used for other purposes than testing and research.
- When this RM is used at a temperature above room temperature, it must be kept in the vacuum or non-oxidizing ambience. At a temperature of 800 °C or higher, a slight amount of clouding and/or black powder was sometimes observed around the position to hold a sample of this RM, which was considered to be attributed to sublimation.
- If mechanical processing (cutting, polishing, etc.) generate cracks and/or distortions, the value of thermal diffusivity deviates from the reference value, which results in deviation of the thermal conductivity from the indicative value.
- If something adheres or is adsorbed to this RM, even if there are no cracks and/or distortions due to mechanical processing

(cutting, polishing, etc.), it is empirically known that the value of thermal diffusivity changes. The value of thermal diffusivity may be brought back to normal when the RM is appropriately cleaned/dried and then annealed at a temperature of 800 °C or higher in the vacuum or with non-oxidizing gas being fed for three hours or more.

Precautions for Handling

The procedure to handle common solid graphite should be applied. Refer to the safety data sheet (SDS) on this RM before use.

Preparation

The base material of this RM was ten rods of the commercially-available isotopic graphite (IG-110 manufactured by Toyo Tanso, Co., Ltd.). Each rod was 10 mm in diameter and 100 mm in length. From these rods, disc-shaped pieces with a diameter of 10 mm and a thickness of 1.0 mm and 2.0 mm were prepared by means of machining. The pieces were annealed at a temperature of 800 °C or higher with argon gas being fed for three hours or more at NMIJ.

Technical Information

The reference values and their uncertainties of the thermal diffusivity α and the specific heat capacity c_p used to calculate the indicative value of thermal conductivity of this RM at typical temperature are given in the table below. The uncertainties of the reference values, U_α and U_{cp}, are relative expanded uncertainties which are determined by using relative combined standard uncertainties and coverage factor $k = 2$. They represent the half width of the interval which is presumed to feature the level of confidence of about 95%.

Temperature	Thermal diffusivity		Specific heat capacity	
T (K)	α (m^2s^{-1})	U_α (%)	c_p ($J K^{-1} g^{-1}$)	U_{cp} (%)
300	9.68×10^{-5}	8.8	0.731	4.3
400	6.21×10^{-5}	7.5	0.999	3.7
500	4.57×10^{-5}	6.9	1.220	3.3
600	3.63×10^{-5}	6.7	1.400	3.1
700	3.02×10^{-5}	6.8	1.542	3.1
800	2.60×10^{-5}	6.8	1.650	3.3
900	2.29×10^{-5}	6.8	1.729	3.7

The thermal diffusivity α and the specific heat capacity c_p in the table were calculated by using the following equations:

$$\alpha/(\mathrm{m^2 s^{-1}}) = -3.795\times10^{-5} + 4.091\times10^{-5}\cdot\exp\left(\frac{3.576\times10^{2}}{T/\mathrm{K}}\right)$$

$$c_p(T) = -0.39119 + 4.6281\times10^{-3}(T/\mathrm{K}) - 3.1676\times10^{-6}(T/\mathrm{K})^2 + 7.1481\times10^{-10}(T/\mathrm{K})^3$$

Where: T = temperature

The applicable range of temperatures: 300 K $\leqq T \leqq$ 900 K

The bulk density ρ at room temperature: 1782 kg m^{-3} (with uncertainty of 13.2 kg m^{-3} (k=2))

It has been demonstrated that the thermal diffusivity and the specific heat capacity of this RM remain unchanged within the range of the uncertainties when the heat cycle between room temperature and 800 °C is repeated up to ten times or when the cumulated duration of exposure to high-temperature environment (up to 800 °C) is up to 60 hours. Accordingly, the stability of the thermal conductivity of this RM, which is a product of the thermal diffusivity and the specific heat capacity, is also demonstrated under the same conditions. Within the temperature range from room temperature to maximum allowable temperature for use, no violent reactions were observed between this RM in its specified ambience of use and α-alumina (Al_2O_3) or boron nitride (BN).

NMIJ Analysts

The technical manager for this RM is YAMADA N.; the production manager is AKOSHIMA M., and the analyst is ABE H.

Information

If substantive technical changes occur that affect the value assignment before the expiration of this report, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this RM can be obtained from the contact details given below.

Reproduction of Report

In reproducing this report, it should be clearly indicated that the document is a copy.

April 1, 2020

ISHIMURA Kazuhiko
President
National Institute of Advanced Industrial Science and Technology

If you have any questions about this RM, please contact:
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Revision history
March 2, 2014: The limit of validity of the report was extended from “March 31, 2015” to “March 31, 2020.”
April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”
February 20, 2019: The limit of validity of the report was extended from “March 31, 2020” to “March 31, 2025.”