National Institute of Advanced Industrial Science and Technology

# National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5809-a No. +++



# Quartz Glass for Thermal Diffusivity Measurement

This certified reference material (CRM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in the calibration and validation of instruments used for thermal diffusivity measurement.

# **Certified Values**

The certified values for thermal diffusivity  $\alpha$  of this CRM are given by the following equations. The relative expanded uncertainty U is given below. The uncertainty U is the half-width of the relative expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

$$\alpha/(m^{2}s^{-1}) = 1.647 \times 10^{-6} - 5.008 \times 10^{-9} \cdot (T/K) + 1.027 \times 10^{-11} \cdot (T/K)^{2}$$
$$-9.592 \times 10^{-15} \cdot (T/K)^{3} + 3.401 \times 10^{-18} \cdot (T/K)^{4}$$
$$U/\% = 5.2$$

at  $300 \text{ K} \leq T \leq 800 \text{ K}$ .

The calculated results from the above equations at typical temperature points of this CRM are shown in the table below.

	Temperature <i>T /</i> K	Thermal diffusivity $\alpha / (m^2 s^{-1})$	Relative expanded uncertainty U / %
	300	8.37×10⁻ <sup>7</sup>	5.2
	400	7.60 × 10⁻ <sup>7</sup>	
	500	7.24 × 10 <sup>-7</sup>	
	600	7.08×10⁻ <sup>7</sup>	
	700	7.00 × 10⁻7	
	800	6.95 × 10⁻7	

## Analysis

The certified values (thermal diffusivity) were determined by using the laser flash method. The function (quartic polynomial) as the temperature dependence of thermal diffusivity was determined by least squares fitting for measured thermal diffusivity values of 11 specimens sampled. The specimens for the laser flash thermal diffusivity measurement were coated by gold sputtering and carbon spray. The intrinsic thermal diffusivity at the steady temperature was obtained as a value extrapolated to zero-pulsed heating energy in the plot of apparent thermal diffusivity versus the temperature rise which depends on the pulsed heating energy. Each apparent thermal diffusivity was calculated from a temperature rise curve by analysis with the heat loss model. The thermal diffusivities were calculated from the specimen thickness which was measured by a linear gauge at room temperature.

The relative expanded uncertainty was estimated from the uncertainty components derived from the thermal diffusivity measurements (including uncertainty caused by coating), the determination of the certified values as a function of temperature, stability and homogeneity.

# Date of Shipment: Xxxxx XX, 20XX

#### **Metrological Traceability**

Thermal diffusivity, derived from specimen thickness and heat diffusion time, was determined by the primary measurement system for thermal diffusivity in NMIJ. The primary measurement system in NMIJ was calibrated by block gauges, function generator and R-type thermocouple, all of which were calibrated in a way to be traceable to the International System of Units (the SI). The certified values (thermal diffusivity), therefore, are traceable to the SI.

#### **Expiration of Certification**

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

#### Sample Form

This CRM consists of four disks with a diameter of 10 mm and thicknesses of 0.5 mm, 1.0 mm, 1.5 mm, and 2.0 mm. These four disks are kept in a plastic container.

#### Homogeneity

The homogeneity of this CRM was determined by the variance analysis of the thermal diffusivity values at 300 K of 45 specimens chosen from among the total 468 specimens. The homogeneity has been incorporated in the uncertainty of the certified value. This CRM is homogeneous within the uncertainty of the certified value.

#### Instructions for Storage

This CRM should be stored at temperatures of 23 °C±10 °C and relative humidity of 50 % or less

#### Instructions for Use

• Heat diffusion time of this CRM coated with metal and carbon should be measured by flash methods with metallic and carbon coatings. The thermal diffusivity should be calculated using specimen thickness measured at room temperature with the resolution of 1 µm before coating. This CRM should be handled in vacuum or under inert gas flow (Ar, etc.) above room temperature.

• This CRM should not be used if it is damaged; e.g. cracked and strained.

• This CRM should not be used if some materials are attached to it because such attached materials may affect its thermal diffusivity.

#### **Precautions for Handling**

This CRM should be handled in a similar way to solid-state quartz glass. Refer to the safety data sheet (SDS) on this material before use.

## Preparation

Specimens of this CRM were prepared from 9 wafers with a diameter of 76.20 mm. Thickness of the nine wafers was varied to 0.5 mm, 1.0 mm, 1.5 mm and 2.0 mm. From each wafer, 13 specimens were cut out in machining process.

#### **Technical Information**

• In the measurement presented in this certificate, in order to prevent the light transmission, the CRM was treated with gold coating and carbon spray coating.

• This CRM does not degrade even after going through five thermal cycles from 300 K to 800 K in vacuum.

Reactivity to typical heat-resistant materials: This CRM does not react severely with  $Al_2O_3$  or BN in vacuum at temperatures of room temperature to 800 K.

#### **NMIJ** Analysts

The technical manager for this CRM is YAMADA N., the production manager is AKOSHIMA M., and the analyst is LI M.

#### Information

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

### **Reproduction of Certificate**

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

April 1, 2020

President

ISHIMURA Kazuhiko

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact: National Institute of Advanced Industrial Science and Technology, National Metrology Institute of Japan, Center for Quality Management of Metrology, Reference Materials Office, 1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan Phone: +81-29-861-4059; Fax: +81-29-861-4009, https://unit.aist.go.jp/nmij/english/refmate/