National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate NMIJ CRM 5807-a No. +++



Al₂O₃—TiC Ceramics 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 for thermal diffusivity measurements for example, the flash method.

Certified Value

The certified value for thermal diffusivity α and the relative expanded uncertainties U of this CRM are given from the following equations. The uncertainty U 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%.

$$\alpha/(m^2s^{-1}) = 7.505 \times 10^{-7} + 9.589 \times 10^{-7} \cdot exp\left(\frac{6.635 \times 10^2}{(T/K)}\right)$$
 (300 K $\leq T \leq 1000$ K)
 $U = 6.1\%$ (300 K $\leq T < 380$ K)
 $U = 5.1\%$ (380 K $\leq T < 593$ K)
 $U = 6.1\%$ (593 K $\leq T < 758$ K)
 $U = 7.1\%$ (758 K $\leq T < 921$ K)
 $U = 8.1\%$ (921 K $\leq T \leq 1000$ K)

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

| T/K | α / (m ² s ⁻¹) | U/% |
|------|--|-----|
| 300 | 9.51×10 ⁻⁶ | 6.1 |
| 400 | 5.79×10 ⁻⁶ | 5.1 |
| 500 | 4.37×10 ⁻⁶ | 5.1 |
| 600 | 3.65×10 ⁻⁶ | 6.1 |
| 700 | 3.22×10 ⁻⁶ | 6.1 |
| 800 | 2.95×10 ⁻⁶ | 7.1 |
| 900 | 2.75×10⁻ ⁶ | 7.1 |
| 1000 | 2.61×10 ⁻⁶ | 8.1 |

Analysis

The certified values (thermal diffusivity) were determined using the laser flash method.

99 specimen-sets of the reference material were prepared from one plate of Al_2O_3 —TiC ceramics (100 mm \times 100 mm \times 10 mm). The specimen-sets consisted of three disk-shaped specimens 10 mm in diameter and 1, 2, and 3 mm in thickness, respectively. Nine specimens (two 1-mm-thick disks, five 2-mm-thick disks, and two 3-mm-thick disks) were sampled for determination of the thermal diffusivity of this CRM. Thermal diffusivities at 300 K, 500 K, 640 K, 760 K, 880 K and 1000 K of the 9 specimens were measured in vacuum. The thermal diffusivity as a function of temperature of this CRM was determined by the least-squares fitting to all measured thermal diffusivities of the 9 specimens.

Metrological Traceability

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

Expiration of Certification

This certificate 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 certificate.

Sample Form

This CRM consists of three disks with diameters of 10 mm and respective thicknesses of 1 mm, 2 mm and 3 mm. The three disks are kept in a plastic container.

Homogeneity

The homogeneity of this CRM was estimated as the standard deviation of the thermal diffusivity values at 300 K of 21 specimens 2 mm thick selected from among 99 specimens. This CRM is considered homogeneous within U since the homogeneity is reflected in the uncertainty of the certified value α .

Instructions for Storage

This CRM should be stored at a temperature of 23 °C \pm 10 °C and a relative humidity of 50 % or less

Instructions for Use

- This CRM is to be used only for testing and experimental studies.
- This CRM can be measured without the black coating for the flash method. Measure the thickness of each specimen with a 1 μm resolution at room temperature before use, and calculate the thermal diffusivity using the thickness.
- Use this CRM in vacuum or inert gas flow (Ar, etc.) above room temperature.
- · 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.

Precautions for Handling

Handling of this CRM is similar to that of solid-state ceramics. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

These CRM specimens were prepared from one plate (100 mm \times 100 mm \times 10 mm) of NPA-2 produced by Nippon Tungsten CO., Ltd. NPA-2 is a grade of Al₂O₃—TiC ceramics.

Technical Information

This CRM did not deteriorate during five cycles of heating and cooling between 300 K and 1000 K.

Reactivity to typical heat-resistant materials: There was no reaction with α -Alumina (Al₂O₃) and BN in the temperature range from room temperature to 1000 K and in vacuum or inert gas flow.

NMIJ Analysts

For this CRM, the technical manager is YAMADA N., and the production manager and analyst are AKOSHIMA M.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the

registered customer. 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

ISHIMURA Kazuhiko
President
National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:

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National Metrology Institute of Japan,

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Revision history

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."