

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Report

NMIJ RM 1101-a  
No. +++

## Thermal Expansion Reference Material (Single-Crystal Silicon): Form 2

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 push-rod dilatometers and thermomechanical analyzers or as a reference specimen in thermal expansion measurements.

**Indicative Values**

The indicative values of thermal expansivity  $\alpha$  and their expanded uncertainties  $U$  at typical temperature points for this RM are given in the table below. The uncertainty of each indicative value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor ( $k$ ) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Temperature $T$ (K)	Indicative value $\alpha$ ( $10^{-6} \text{ K}^{-1}$ )	Expanded uncertainty $U$ ( $10^{-6} \text{ K}^{-1}$ )
293.15	2.551	0.028
300	2.612	0.028
350	2.978	0.027
400	3.244	0.026
450	3.446	0.026
500	3.606	0.025
550	3.736	0.025
600	3.846	0.025
650	3.938	0.026
700	4.017	0.026
750	4.085	0.026
800	4.144	0.026
850	4.198	0.026
900	4.248	0.027
950	4.296	0.027
1000	4.335	0.027

The indicative values of thermal expansivity and the corresponding expanded uncertainties given in the above table are obtained using the following equation:

$$\alpha(T) / (10^{-6} \text{ K}^{-1}) = -6.4822 + 7.1401 \times 10^{-2} \cdot (T / \text{K}) - 2.2935 \times 10^{-4} \cdot (T / \text{K})^2 + 4.1952 \times 10^{-7} \cdot (T / \text{K})^3 - 4.4111 \times 10^{-10} \cdot (T / \text{K})^4 + 2.4831 \times 10^{-13} \cdot (T / \text{K})^5 - 5.7954 \times 10^{-17} \cdot (T / \text{K})^6.$$

This equation is valid in the temperature range of 293.15 K to 1000 K.

### Analysis

Each indicative value of this RM was determined based on absolute measurements of thermal expansivity for six silicon samples. The six test samples were cut from a silicon single-crystal rod with a diameter and length of 60 mm and 125 mm, respectively, which was grown by the floating zone melting method. All distributed pieces of this RM were also produced from this silicon rod. According to the high-temperature thermal expansion measurement manual (QMC TE02A), the thermal expansivity of the six test samples was measured by a laser interferometric dilatometer in the following way:

Each test sample was heated and cooled in a stepwise manner over the temperature range of 293 K to 1050 K. The thermal expansivity of the test sample was calculated as

$$\alpha(T) = \frac{L_{n+1} - L_n}{L_0 (T_{n+1} - T_n)}$$

where  $L_0$  is length of a sample at room temperature (293.15 K), ( $T_n$  and  $T_{n+1}$ ) are the adjacent temperatures of the sample in the stepwise heating and cooling cycle, and ( $L_n$  and  $L_{n+1}$ ) are the lengths of the sample at  $T_n$  and  $T_{n+1}$ . The value of  $L_0$  was measured by a digital linear scale. The temperature difference ( $T_{n+1} - T_n$ ) was set to be approximately 50 K or 25 K. For each sample, 64 values of thermal expansivity were obtained. All thermal expansivity values of the six test samples were pooled, and the sixth-order polynomial function of temperature was obtained by the method of least squares. The samples were heated in a furnace in which helium gas at an amount equivalent to a pressure of 95 Pa and a temperature of 293 K was sealed during the measurements.

### Expiration of Report

The report of this RM is valid until March 31, 2030, provided that the material remains unopened and is stored in accordance with the instructions given in this report.

### Description of the Material

This RM is in the form of a block 9 mm square and 60 mm long.

### Homogeneity

The homogeneity of the RM was determined by analyzing the measurement results of the six test samples taken from different positions of the silicon single-crystal rod. The homogeneity of thermal expansivity has been incorporated into the uncertainties of the indicative values.

### Instructions for Storage

This RM should be stored at a temperature of  $23\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$  and at a relative humidity of 50 % or less.

### Instructions for Use

It is recommended that the thermal expansion along the 60 mm longitudinal direction of the distributed piece of the RM is used as a reference for calibration or verification of a dilatometer, though thermal expansion characteristic of silicon is isotropic. This is because the indicative values given above correspond to the thermal expansivity measured along the longitudinal direction. A distributed piece of the RM should be heated in vacuum or in an atmosphere of inert gas such as argon or nitrogen.

### Precautions for Handling

This RM is considered as a chemically safe material under normal conditions for use. When handling, wear appropriate protective gloves. Refer to the safety data sheet (SDS) on this RM before use.

### Preparation

This RM was made from high-purity single crystal silicon produced by the floating zone melting method. Each distributed piece of the RM, produced from the silicon ingot, is in the form of a block 9 mm square and 60 mm long.

### NMIJ Analysts

The technical manager for this RM is YAMADA N. The production manager and analyst are WATANABE 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 CRM, please contact:  
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#### Revision history

April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”  
September 19, 2019: The expiration of report was extended from March 31, 2020 to March 31, 2025.  
November 24, 2023: The expiration of report was extended from March 31, 2025 to March 31, 2030.