National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Certificate

NMIJ CRM 5803-a (Shape 2)
No. +++

Single-Crystal of Silicon for Thermal Expansivity Measurement (at Cryogenic Temperature)

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

Certified Values

The certified values of thermal expansivity α and their expanded uncertainties U in this CRM are given from the following equations. The value of α is the linear thermal expansivity based on a specimen length at 20 °C (L_0). The value of U given by the equation 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 %.

$$\alpha/(10^{-6}\,\mathrm{K}^{-1}) = \begin{cases} -5.081338\times10^{-5}\cdot(T/\mathrm{K})^2 + 1.142423\times10^{-5}\cdot(T/\mathrm{K})^3 \\ \qquad -6.167704\times10^{-7}\cdot(T/\mathrm{K})^4 + 7.451275\times10^{-9}\cdot(T/\mathrm{K})^5 \ \text{at}\ 20\,\mathrm{K}\le T<35\,\mathrm{K} \\ -4.486603\times10^{-1} + 4.958959\times10^{-2}\cdot(T/\mathrm{K}) - 1.794136\times10^{-3}\cdot(T/\mathrm{K})^2 \\ \quad +2.260789\times10^{-5}\cdot(T/\mathrm{K})^3 - 1.179978\times10^{-7}\cdot(T/\mathrm{K})^4 + 2.280928\times10^{-10}\cdot(T/\mathrm{K})^5 \ \text{at}\ 35\,\mathrm{K}\le T<125\,\mathrm{K} \\ 3.157177 - 1.165070\times10^{-1}\cdot(T/K) + 1.272514\times10^{-3}\cdot(T/K)^2 \\ \quad -5.672490\times10^{-6}\cdot(T/\mathrm{K})^3 + 1.200980\times10^{-8}\cdot(T/\mathrm{K})^4 - 9.973051\times10^{-12}\cdot(T/\mathrm{K})^5 \ \text{at}\ 125\,\mathrm{K}\le T\le300\,\mathrm{K} \end{cases}$$

$$U/(10^{-6}\,\mathrm{K}^{-1}) = 5.1\times10^{-3} - 2.2\times10^{-2}\cdot(T/\mathrm{K})^{-1} + 1.6\cdot(T/\mathrm{K})^{-2} \ \text{at}\ 20\,\mathrm{K}\le T\le300\,\mathrm{K}$$

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

Temperature T (K)	Certified value α (10^{-6} K^{-1})	Expanded uncertainty U (10^{-6} K^{-1})	Temperature T (K)	Certified value α (10^{-6} K^{-1})	Expanded uncertainty U (10^{-6} K^{-1})
20	-0.0038	0.0080	170	0.8724	0.0050
30	-0.0558	0.0061	180	1.0563	0.0050
40	-0.1675	0.0056	190	1.2329	0.0050
50	-0.2947	0.0053	200	1.4007	0.0050
60	-0.4008	0.0052	210	1.5593	0.0050
70	-0.4639	0.0051	220	1.7086	0.0050
80	-0.4745	0.0051	230	1.8487	0.0050
90	-0.4319	0.0051	240	1.9803	0.0050
100	-0.3420	0.0050	250	2.1039	0.0050
110	-0.2143	0.0050	260	2.2202	0.0050
120	-0.0594	0.0050	270	2.3297	0.0050
130	0.1141	0.0050	280	2.4328	0.0050
140	0.2995	0.0050	290	2.5294	0.0050
150	0.4907	0.0050	300	2.6190	0.0050
160	0.6829	0.0050			

Analysis

The certified value of this CRM was determined in the following way:

(1) The thermal expansion was measured by a laser interferometric dilatometer, and the specimen length was measured by a digital linear scale at 20 °C.

(2) The certified values were determined as a function of temperature by performing the fitting of the measured thermal expansion values with the restricted least squares method which used the measurement uncertainty as a weight.

Metrological Traceability

Each certified value (α) is determined based on the measurement results of a change of specimen temperature (ΔT), a change of specimen length (ΔL), and a specimen length at 20 °C (L_0). The values of ΔT, ΔL, and L_0 are measured by the instruments calibrated by the reference standards, namely an Iodine-stabilized He Ne laser, a Platinum resistance thermometer, a standard resistance, and a block gauge. The certified value, therefore, is traceable to the International System of Units (SI).

Expiration of Certification

This certificate is valid from the date of shipment to March 31, 2030, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is in the form of a rectangular block 10 mm square and 60 mm long. The CRM is kept in a plastic container.

Homogeneity

The homogeneity of the CRM was determined by analyzing the measurement results on four specimens (20 mm × 20 mm× 8 mm) which were cut from different positions with respect to the axial and radial directions of a cylindrical ingot of single-crystal silicon. The homogeneity of thermal expansivity has been incorporated into the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored at a temperature of 35 °C or less under the nitrogen gas atmosphere.

Instructions for Use

This CRM should not be used for any other purposes than the calibration of a dilatometer.

Cautions with respect to thermal cycles:

- Avoid a thermal shock which causes cracks in the CRM.
- Avoid heating to 315 K or more.

Processing of specimens:

- Users can cut and polish the CRM to adjust it to their dilatometers.
- When processing, note that the certified value represents the thermal expansivity in the 60 mm longitudinal direction of the distributed CRM.
- When processing, care should be taken to avoid crack generation and plastic deformation in a specimen by giving consideration to thermal and mechanical stress.
- Do not use a specimen in which cracks are generated.

Precautions for Handling

Handle this CRM in a similar way to solid-state silicon. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

This CRM was prepared from a high-purity single-crystal silicon ingot produced by the floating zone melting method in Shin-Etsu Chemical Co., Ltd. The CRM was produced by cutting the silicon ingot into the form of rectangular block 10 mm square and 60 mm long.

NMIJ Analysts

The technical and production managers for this CRM are YAMADA N. and the analyst is YAMADA N.

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.

Note

This CRM is the same as NMIJ RM 1103-a supplied from fiscal 2007. NMIJ RM 1103-a was replaced by NMIJ CRM 5803-a, single-crystal silicon for thermal expansivity measurement (at cryogenic temperature), in February 2011.

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
March 19, 2014: The expiration of this certificate 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.”
September 4, 2018: The limit of validity of the certificate was extended from “March 31, 2020” to “March 31, 2025.”
September 28, 2023: The limit of validity of the certificate was extended from “March 31, 2025” to “March 31, 2030.”