

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 5605-a  
No. +++

## Hafnium Oxide Film for Quantitative Analysis of Hafnium

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 precision control of analysis as well as in confirmation of the measurement procedure and results for quantifying hafnium in a hafnium oxide film by means of Rutherford backscattering spectrometry, inductively coupled plasma-mass spectrometry (ICP-MS), and X-ray fluorescence analysis.

**Certified Value**

The certified value of hafnium for this CRM is given in the table below. The uncertainty of the certified 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 %.

Element	Certified value, Area density ( $\mu\text{g}/\text{cm}^2$ )	Expanded uncertainty, Area density ( $\mu\text{g}/\text{cm}^2$ )
Hf	3.59	0.09

**Analysis**

The certified value is hafnium mass divided by graphically measured area of the film side of the CRM. The hafnium mass was determined as an arithmetic mean of the measured values from isotope dilution (ID) - ICP-MS and instrumental neutron activation analysis (INAA).

**Metrological Traceability**

The amount of hafnium was determined using NIST SRM 3122 (Hafnium Standard Solution) as calibration standard while the area of the sample surfaces was determined traceably to JCSS calibrated standard scales for length. The certified value is traceable to the International System of Units (SI).

**Expiration of Certification**

This certificate is valid for one year from the date of shipment, provided that the material is stored in accordance with the instructions given in this certificate.

**Sample Form**

The size of this CRM is a 10 mm square tip with a 0.5 mm thickness and the CRM is kept in a chip tray in a plastic vessel.

**Homogeneity**

Homogeneity of the hafnium concentration in the CRM was determined through the ID-ICP-MS analysis for 9 pieces taken from 96 chips, cut of three hafnium oxide films fabricated on silicon wafers in 100 mm diameters. The homogeneity is reflected in the uncertainty of the certified value.

**Instructions for Storage**

This CRM should be stored at a temperature between 5 °C and 40 °C in a clean atmosphere and shielded from direct sunlight. Storage in dry air or nitrogen gas is recommended.

**Instructions for Use**

The hafnium oxide film is deposited on the mirror polished side of the CRM. The CRM is placed with the film side up in the chip tray. The surface should be treated carefully to avoid any damage or contamination of the film. The certified value is determined for whole area of the chip. If the sampling spot is much smaller than the chip size, several points on the film surface must be picked up at the time of analysis and the average value of the analysis should be adopted.

**Precautions for Handling**

Refer to the safety data sheet (SDS) on this CRM before use.

**Preparation**

Hafnium oxide film was deposited on one side of a 100 mm $\phi$  single crystal silicon (100) wafer by magnetron sputtering. The CRM was prepared by cutting the deposited wafers into 10 mm  $\times$  10 mm squares in size. The film deposition, the cutting and the packaging were performed by NTT Advanced Technology Corporation.

**NMIJ Analysts**

The technical manager for this CRM is ITO K., the production manager is TAKATSUKA T., and the analysts are TAKATSUKA T., HIRATA K., NONOSE N., and MIURA T.

**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**

Determination of the hafnium mass by INAA was performed by using facilities of the Research Reactor Institute, Kyoto University.

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."  
November 22, 2016: The description in "Expiration of Certification" was changed to "one year from the date of shipment."