National Institute of Standards & Technology

Certificate

Standard Reference Material® 1995

Standard Sapphire Single Crystal Wafer for Crystalline Orientation

This Standard Reference Material (SRM) is intended for use in the calibration of instruments (X-ray diffractometers) used to measure the crystal orientation of wafers relative to the crystal surface. The SRM unit consists of a 50 mm diameter sapphire wafer.

Certified Values and Uncertainites: NIST certified values are values for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1]. The crystal orientation of the sapphire crystal C-axis (001) relative to the surface normal has been measured both parallel and perpendicular to an edge flat (A-axis (11-20)) that is manufactured into the wafer. The statistical uncertainty (type A) has been combined with the maximum systematic error (type B) in quadrature to determine an overall error for all wafers of 8.4 arc seconds. The certified crystal orientation is found in Table 1. The calibration uncertainty components are listed in Table 2.

Expiration of Certification: The certification of this SRM is deemed to be indefinite within the stated uncertainties, provided the SRM is stored and handled in accordance with the "Storage and Handling" and "Instructions for Use" sections of this certificate. However, certification will be nullified if the SRM is contaminated or otherwise altered.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination for this SRM was provided by E.C. Benck and R.J. Matyi of the NIST Atomic Physics Division.

The crystal orientation measurement technique, development, and certification of this SRM were performed by E.C. Benck of the NIST Atomic Physics Division.

Statistical analysis was performed by B. Toman of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Carl J. Williams, Chief
Atomic Physics Division

Gaithersburg, MD 20899
Certificate Issue Date: 02 October 2008

Robert L. Watters, Jr., Chief
Measurement Services Division

Storage and Handling: Sapphire is typically inert under ambient and testing conditions. The sapphire wafer may be returned to the wafer storage box and reused. The wafer should not be dropped or similarly mechanically stressed.

INSTRUCTIONS FOR USE

Dust should be removed from the SRM wafer surface and the wafer mount prior to installation in the X-ray diffractometer. The flat manufactured into the wafer should be mounted either parallel or perpendicular to the X-ray plane of dispersion with the polished surface facing towards the X-ray source. Consult the equipment manufacturer's operating instructions for performing the actual measurements with the SRM. Measurements should be made near the center of the wafer.

Summary of Analytical Method: The angular measurements were made using a high resolution double crystal X-ray diffractometer. The diffractometer utilizes a dispersive geometry with a fixed first crystal; for this work, a (004) reflection from a highly perfect silicon reference crystal is used. The sample crystal is mounted on an Ultradex[1] precision indexing table that is in turn mounted on a high precision rotation spindle that is driven by a stepper motor and non-rotating micrometer via a sine arm. The inclusion of the Ultradex table allows the sample to be indexed in 1° increments for large excursions while leaving the sine arm drive (total angular range of approximately ± 3°) for precision movements.

Attached to the sample rotation spindle is a polarization-encoded optical angle interferometer that employs a calibrated (NIST-traceable) He-Ne laser source. Angle measurements were performed by counting interference fringes with a calibration technique relating fringes to angle increment that was established by conventional angle metrology methods. Since the wavelength of the interferometer is traceable to the SI, the measurements of angular misorientation are as well.

The following measurement procedure was used for each of the sapphire wafers in the production run:

1. The wafers were used as supplied by the vendor with no additional preparation.
2. The sample was mounted on a precision rotation axis (ϕ-axis) with the wafer flat parallel to the plane of dispersion. The sample and ϕ-axis are mounted on the sample rotation or ω-axis of a double crystal X-ray diffractometer. The X-ray beam nominally samples a 1 cm wide area at the center of the wafer. The ω-axis is perpendicular to the X-ray plane of dispersion; the rotation of this axis will be calibrated using circle closure with an optical polygon and a precision nulling autocollimator. Angles were measured using a polarization-encoded angle interferometer. These attributes (closure calibration and angle measurement with an angle interferometer with calibrated laser) ensure traceability to the SI.
3. The sample surface was aligned with respect to a two-axis electronic autocollimator. The sample surface was made perpendicular to the autocollimator to a precision of better than 2×10^{-4} radians (i.e., better than 4 angular seconds). The angle in the plane of dispersion corresponding to the crystal being aligned with the autocollimator is determined from a linear least squares fit to a series of autocollimator signal measurements versus angle measurements.
4. The reflection from the wafer was recorded with $CuK\alpha_1$ radiation monochromated by a highly perfect silicon reference crystal. The angle was determined using a curve fitting routine with the X-ray detector signal and angle measurements. The angle difference between the autocollimator signal and the X-ray reflection was calculated.
5. The sample was rotated 180° about the ϕ-axis. Steps (3) and (4) were repeated in order to determine the surface normal and reflection angles from which the difference angle was calculated.
6. The angle difference between the autocollimator and the X-ray reflection determined in (4) and (5) was summed, and one half of this value is the wafer misorientation with respect to the surface about an axis perpendicular to the wafer flat.
7. The sample was rotated 90° to orient the wafer flat perpendicular to the plane of dispersion.
8. Steps 3, 4, and 5 were repeated.
9. The angle differences between the autocollimator and the X-ray reflection determined in step 8 were summed, and one half of this value is the wafer misorientation with respect to the surface about an axis parallel to the wafer flat.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate to adequately describe the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are the best available for the purpose.

Table 1. Certified Crystal Orientation

Wafer Label	Angle Parallel to Flat		Angle Perpendicular to Flat	
	Arc Minutes	Arc Seconds	Arc Minutes	Arc Seconds
AA_004	-24	-37.1	-2	-3.3
AA_005	-25	-36.4	1	59.7
BB_001	-3	-38.9	1	59.4
BB_002	1	58.5	3	32.6
BB_003	0	20.4	3	7.6
BB_006	2	1.4	-3	-18.6
BB_007	-1	-19.2	0	13.5
BB_008	0	29.6	-3	-11.7
BB_009	0	46.3	0	41.4
BB_010	-1	-37.4	0	-7.3
BB_012	2	15.9	-2	-51.0
BB_013	0	53.7	0	53.2
BB_015	2	36.3	2	36.6
CC_001	0	29.2	-3	-40.4
CC_004	0	-4.1	3	24.2
CC_005	3	9.2	-1	-42.9
CC_006	2	0.0	-3	-24.3
CC_007	0	29.5	-1	-31.3
CC_008	0	20.8	3	13.9
CC_009	-3	-28.7	1	46.7
CC_010	0	30.0	-3	-16.0
CC_011	1	57.8	3	47.5
CC_014	-3	-17.0	-2	-16.5
CC_015	0	23.2	3	16.5
CC_017	0	34.5	-1	-26.0
CC_019	0	6.8	3	6.3
CC_020	-3	-36.1	-2	-7.4
CC_021	5	20.9	2	32.8
CC_022	2	17.4	-3	-36.7
CC_024	2	14.1	3	27.5
CSI_3060103	0	16.2	3	11.0
CSI_3060104	-1	-14.0	0	35.1
CSI_3060106	1	57.8	3	30.7
CSI_3060107	1	11.7	0	35.9
CSI_3060108	5	15.6	2	47.3
CSI_3060109	0	23.3	1	16.1
CSI_3060111	0	35.2	0	-59.0
CSI_3060112	0	28.1	1	36.5
CSI_3060113	-4	-6.4	2	4.3
CSI_3060114	0	23.7	0	42.4
CSI_3060115	0	19.0	-3	-22.5
CSI_3060116	2	25.4	3	29.2
CSI_3060117	0	20.7	1	33.2
CSI_3060120	-1	-25.4	0	-3.5

Table 1. Crystal Orientation (cont.)

Wafer Label	Angle Parallel to Flat		Angle Perpendicular to Flat	
	Arc Minutes	Arc Seconds	Arc Minutes	Arc Seconds
CSI_3060122	0	29.4	-1	-30.8
CSI_3060123	5	20.4	-2	-36.9
CSI_3060125	0	34.4	0	-59.1
CSI_3060126	5	25.7	-2	-48.4
CSI_3060127	0	13.0	1	6.7
CSI_3060128	0	30.7	-1	-35.4
CSI_3060129	0	17.6	-3	-1.7
CSI_3060131	0	31.4	-1	-23.1
CSI_3060132	0	37.1	3	2.5
CSI_3060133	0	33.5	0	37.2
CSI_3060134	-1	-23.7	0	-31.1
CSI_3060135	-3	-23.7	-1	-54.4
CSI_3060136	0	20.8	-2	-58.8
CSI_3060137	1	54.6	3	34.4
CSI_3060138	1	53.6	-3	-33.4
CSI_3060139	5	13.9	-3	-0.6
CSI_3060140	0	20.9	-3	-25.6
CSI_3060141	0	30.5	3	18.2
CSI_3060142	0	6.3	2	42.8
CSI_3060143	2	35.0	2	30.8
CSI_3060146	0	18.2	-1	-6.7
CSI_3060148	0	31.5	3	5.8
CSI_3060149	-4	-7.4	-1	-58.4
CSI_3060150	0	5.7	-3	-14.3
CSI_3060153	-1	-17.5	0	-16.6
CSI_DD_1364-3	4	41.4	-1	-52.5
CSI_DD_1365-2	5	10.0	1	9.6
CSI_DD_1372	0	47.0	-5	-16.8

Calibration Uncertainties: Calibration uncertainty components are listed in Table 2.

Table 2. SRM 1995 Calibration Uncertainty Components

Error Description	Value (angular seconds)
Misalignment of Surface Normal	negligible
Misalignment of ϕ-Axis	negligible
Mounting Misalignment	0.15
Angular Rotation about ϕ-Axis	.9
Peak Assignment	0.1
Crystal Inhomogeneity	5
Measurement Repeatability	6.7

Misalignment of the Sample Normal: Misalignment of the sample normal out of the plane of the diffractometer by an amount ϕ leads to a tilt error $\Delta\theta = (\phi^2/2) \tan \theta$. The fact that the error depends on the square of the misorientation means that as the sample is rotated 180° about the ϕ-axis, the apparent shift in the diffracted peak position will be the same ($\theta + \Delta\theta$), so by taking the difference, the tilt error cancels out.

Misalignment of the ϕ-Axis: Realignment of the wafer with respect to the autocollimator after each rotation about the ϕ-axis results in errors due to misalignment of the ϕ-axis from the plane of the diffractometer negligible.

Mounting Misalignment: An error in aligning the major flat to the diffractometer plane results in an error in the direction of the crystal misorientation, but not the overall magnitude. Although care was taken to remove any dust or debris from the wafer and the mounting bar, there always is the possibility of dust causing the wafer flat to be misaligned. In addition, there is the possibility of the wafer support itself being misaligned relative to the diffractometer plane resulting in a systematic error. Consequently, we estimate that the absolute accuracy of the wafer alignment to be better than 20 angular seconds. For the magnitude of angles being measured, this corresponds to an uncertainty of 0.15 angular seconds.

Angular Rotation about ϕ-Axis: Errors in the angular positioning about the ϕ-axis result in the most significant uncertainty in the crystal orientation measurements. The angular positioning of the ϕ-axis bearing is determined by a set of interlocking gear teeth. Based on the manufacturer's specifications, the angular accuracy should be better than 4 angular minutes. Consequently, for the magnitude of angles being measured, the maximum systematic uncertainty due to any measured angle is 0.9 angular seconds.

Peak Assignment: Error in the peak assignment, due to statistical noise, is not a significant source of error. The intensity of the X-ray beam was maintained to produce reasonable signal-to-noise ratios. Repeat measurements of the same rocking curve without moving the ϕ-axis maintain results in the same angle measurement to within 0.1 arc second.

Crystal Inhomogeneity: Although the sapphire wafers are of good quality, the wafers still exhibited some variation in the crystal orientation across the surface of the wafer. This is probably due to internal strains within the crystal. A broad X-ray beam was used to average any localized variations in the crystal orientation. These internal strains are probably responsible for the observed line widths of the rocking curves. Crystals, in which the rocking curves were so broad as to blend the two X-ray peaks, were rejected. A few of the crystals produced rocking curves where the X-ray peaks exhibited sub-peaks; this was probably due to localized regions of strain sampled by the X-ray. The curve fitting program could be forced to converge on an individual sub-peak instead of the envelope of the entire peak. In the worst case, the resulting measurements using a sub-peak differed from the normal peak assignment method by 5 arc seconds.

Measurement Repeatability: The statistical measurement uncertainties of SRM 1995 were determined from repeated measurements of three wafers selected from the production run. Based on the statistical measurements, an expanded uncertainty at the 95 % level of confidence was determined to be below 7 arc seconds.

REFERENCE

[1] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.