



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 656

Silicon Nitride Powders (Quantitative Analysis Powder Diffraction Standard)

This Standard Reference Material (SRM) consists of two powders intended for quantitative analysis of the α and β polymorphs of silicon nitride via powder diffraction methods. The powders are combinations of the α and β polymorphs; one is high in the α phase content (α 656), while the other contains a larger amount of the β polymorph (β 656). This selection allows the user to choose the powder which is optimal for their specific analysis. A unit of SRM 656 consists of approximately 10 g of each powder.

Material Description: The powders consist of sub-micrometer, equi-axial, non-aggregated grains that do not display the effects of extinction or preferred orientation. The powders were bottled in an argon atmosphere; silicon nitride is known to slowly react with atmospheric moisture. An analysis of the phase quantification and lattice parameter values from Rietveld analyses of X-ray powder diffraction data indicated that the SRM material was homogeneous with respect to diffraction properties.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account. The measurands are the certified values for the crystalline phase purity of the material (silicon nitride), the mass fractions (expressed as a percent) of the two polymorphs, and the lattice parameters which are provided in Tables 1 and 3. Metrological traceability is to the SI units for the derived unit of mass fraction (expressed as milligrams per kilogram), and for length (expressed as nanometers); for crystalline phase purity and lattice parameters, respectively. The certified values and uncertainties were calculated according to the method described in the ISO/JCGM Guide [1].

Information Values: An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to adequately assess the uncertainty associated with the value, or it is a value derived from a limited number of analyses. Information values cannot be used to establish metrological traceability. The information values for the relative intensities, the Reference Intensity Ratio (RIR) [2] (for a complete discussion of RIR methods, see reference 3) of the two polymorphs (α/β) of silicon nitride, microstructural data from an analysis of line profile broadening and the particle size distribution, as determined by laser scattering, are given in Tables 4, 5, and 6.

Expiration of Certification: The certification of **SRM 656** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Coordination of material preparation, technical measurements, and data analyses for the certification of this SRM was performed by J.P. Cline of the NIST Materials Measurement Science Division and R.B. Von Dreele of Argonne National Laboratory (Argonne, IL). Supplemental characterization was performed by M.D. Vaudin of the NIST Materials Measurement Science Division and D.B. Minor, formerly of NIST.

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Certificate Revision History on Last Page

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Statistical consultation for the original certification was by provided S.B. Schiller and L.M. Oakley formerly of the NIST Statistical Engineering Division. The analyses performed in the 2017 was by J. Filliben of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR STORAGE AND USE

Storage: SRM 656 was bottled in an argon atmosphere to protect against humidity. Silicon nitride is known to react slowly with atmospheric moisture. When not in use, store the unused portion of this powder tightly capped in the original bottle or in a manner with similar or greater protection against humidity, such as a desiccator.

SOURCE, PREPARATION, AND ANALYSIS⁽¹⁾

Materials: The powder used for α 656 was UBE E10, UBE Industries Ltd. (Tokyo, Japan), while that used for β 656 was Denka SN-P21FC, Denki Kagaku Kogyo K.K. (Tokyo, Japan). The high β phase content powder was donated to NIST by Denki Kagaku Kogyo K.K. We also acknowledge the cooperation of UBE Industries Ltd.

Phase Purity: A long-count-time X-ray powder diffraction pattern of a given powder sample may offer data consistent with a high purity powder, i.e., no impurity phases and a background that is consistent with contributions of air scatter from the incident beam and thermal diffuse scatter from the sample. However, the surface region of any crystalline material will not diffract as the bulk due to relaxation of the crystal structure and inclusion of surface reaction products. While this surface layer may only be on the order of a few crystallographic units in thickness, in a finely divided solid it can easily account for several percent of the total mass. The characterization of “phase purity” or “amorphous content” discussed herein is not in the context of a mechanically separable impurity phase, but it is a microstructural characteristic innate to the chemistry of Si_3N_4 and the production history of the SRM feedstock.

Certification Method: This SRM was certified in 1995 with respect to the mass fraction of the α and β polymorphs of Si_3N_4 that exhibited Bragg scattering in correspondence to that of crystalline components, or phase composition and phase purity. The certification procedure utilized Quantitative Rietveld Analyses (QRA) [4] (for a complete discussion of the Rietveld method, see references 5 and 6) of neutron time-of-flight (TOF) powder diffraction measurements in conjunction with the use of SRM 676 [7] as the internal standard. This procedure referenced the phase purity of SRM 656 against that of SRM 676. The basis of the method rests on an analysis of the discrepancy between the results of powder diffraction experiments, which measure the mass of crystalline material exhibiting Bragg diffraction, relative to weighing operations, which include all components. Neutron data are considered to be essentially free of a systematic bias in phase quantification that is often observed in analyses laboratory X-ray powder diffraction data. The phase purity of SRM 656, as certified in 1995, reflected our approximate knowledge of the phase purity of SRM 676 in 1995 [8]; however, SRM 676 was certified with respect to phase purity in 2005. Therefore, the certified phase purity of SRM 656 was revised in 2005. The lattice parameters of SRM 656 were certified in 2017. The original data for determination of the relative intensity and the $\text{RIR}_{\alpha/\beta}$ were also re-analyzed in 2017 using modern profile fitting methods.

The QRA of laboratory, divergent beam X-ray powder diffraction (XRPD) data may display a systematic bias of less than 2 %; however, these results were as precise as those determined from the TOF data. Therefore, while the certified phase composition was determined from the TOF data, the homogeneity of the SRM material was verified with Rietveld analyses of XRPD data. It should be noted that the mechanism inducing this bias in QRA is not operative in RIR based methods. The emission spectrum of Cu $K\alpha$ radiation of the XRPD experiment is used as the basis for constructing the diffraction profiles within the fundamental parameters approach (FPA) [9] method for diffraction line profile analysis. The emission spectrum provides the linkage of the certified lattice parameter values to the fundamental unit of length, as defined by the International System of Units (SI) [10]. With the use of the FPA, diffraction profiles are modeled as a convolution of functions that describe the wavelength spectrum, the contributions from the diffraction optics, and the sample contributions resulting from microstructural features. Analysis of data from a divergent-beam instrument requires knowledge of both the diffraction angle and the effective source-sample-detector distance. Two additional models are therefore included in the FPA analyses to account for the effect of the sample height and attenuation. Certification data were analyzed in the context of both Type A

⁽¹⁾ Certain commercial instruments, materials, or processes are identified in this certificate to adequately specify 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

uncertainties, assigned by statistical analysis, and Type B uncertainties, based on knowledge of the nature of errors in the measurements, to result in the establishment of robust uncertainties for the certified values.

Certification Procedure: Ten bottles of each of the two SRM 656 materials were selected from the populations in accordance to a stratified random protocol. Five randomly selected samples from each material were admixed with a known amounts, 25 % in the case of α 656 and 50 % with β 656, of SRM 676 for TOF neutron diffraction analysis. TOF data were obtained on the High Intensity Powder Diffractometer (HIPD) at the Manuel Lujan, Jr., Neutron Scattering Center (LANSCE, Los Alamos, NM). The samples were contained in 9.5 mm diameter by 50 mm long vanadium cans during the analysis. Each sample was exposed to the neutron beam for 1.68 h to 6.13 h with the LANSCE source operating at approximately 60 μ A proton beam current. Data used for this certification were obtained from detector banks positioned at $\pm 153^\circ 2\theta$, each covering a d-spacing range of 0.04 nm to 0.48 nm.

The Rietveld refinements were performed using the General Structure Analysis System (GSAS) [11] in two global refinements that included the five datasets from the two SRM 656 materials. The structure used for the α phase was obtained from Marchand, *et al.* [12]. That used for the β phase was in correspondence with Billy, *et al.* [13]. The refinements of three phases in these samples included: scale factors, lattice parameters, an absorption factor and six terms of a background function describing the effects of thermal diffuse and incoherent scattering for each data set. The structural parameters and crystallite size and microstrain [14] parameters for the majority phases (alumina and α silicon nitride in the high α material, and alumina and β silicon nitride in the high β material), were refined. The amorphous phase content was determined from the mass fraction ratio determined from the diffraction experiment relative to that of the weighing operation, with the latter ratio being corrected for the known phase purity of SRM 676. The certified phase purity of these materials, and the phase composition with respect to the α and β polymorphs, with $k = 2$ expanded uncertainties, is given in Table 1.

Table 1. Certified phase purity and phase composition for SRM 656.

α 656 (High α Phase Powder)		
Phase	Mass	Uncertainty
α	87.4 %	± 0.03 %
β	3.0 %	± 0.03 %
Amorphous	9.6 %	± 0.45 %
β 656 (High β Phase Powder)		
α	16.3 %	± 0.25 %
β	75.1 %	± 0.25 %
Amorphous	8.6 %	± 2.10 %

Laboratory XRPD data for certification of lattice parameters were collected on a NIST-built diffractometer configured with a Johansson Ge [111] incident beam monochromator (IBM), sample spinner and a linear Si-strip position sensitive detector (PSD). A full discussion of this machine, its alignment and calibration can be found in Cline, *et al.* [15]. The 1.5 kW copper tube of fine focus geometry was operated at a power of 1.2 kW during data collection. The variable divergence incident slit was set to 0.9° . A 1.5° Soller slit was located in front of the PSD window to limit axial divergence, no Soller slits were used in the incident beam. The PSD was scanned using a variable window length and a combination of coarse and fine steps in $\theta/2\theta$ was used that allowed for data to be collected at high resolution in a timely manner [16]. Samples were spun at 0.5 Hz during data collection. The machine was equipped with an automated anti-scatter slit that blocked air scatter from the incident beam from entering the PSD, where it would otherwise contribute to the low angle background level. Data were collected from $18^\circ 2\theta$ to $155^\circ 2\theta$; the scan time was approximately 2.5 hours. The machine was located within a temperature-controlled laboratory space where the nominal short-range control of temperature was $\pm 0.1^\circ \text{K}$. The temperature was monitored using two 10 k Ω thermistors with a Hart/Fluke BlackStack system that was calibrated at the NIST temperature calibration facility [17] to $\pm 0.002^\circ \text{C}$. The source was equilibrated at operating conditions for at least an hour prior to recording any certification data.

The certification data were analyzed using the FPA method with Rietveld analyses as implemented in TOPAS [18]. Mendenhall, *et al.* [19] verified that TOPAS operated in accordance with published models for the FPA. The analysis used energies of the Cu $K\alpha_1$ emission spectrum as characterized by G. Hölzer, *et al.* [20]. The refined parameters included the scale factors, Chebyshev polynomial terms for modeling of the background, the lattice parameters, specimen displacement and attenuation terms, structural parameters and a term for Lorentzian strain broadening.

The crystallite size broadening was modeled with a log-normal size distribution of spherical crystallites using the Scardi and Leoni formalism [21]. A hexagonal crystallite shape model was used for the β phase of the β 656 data. A discussion of the incident spectrum from the IBM and the approach used in its modeling, as well as the characterization of the instrument profile function (IPF) via the FPA are discussed in Cline *et al.* [15]. The procedure used high-count-time data collected from SRM 660b [22,23] using a relatively small divergence slit angle of 0.5° . The incident spectrum was then modeled with a refinement of the breadths and intensities of three Gaussian profiles at the $K\alpha_{11}$ location, as defined by Hölzer, and a fourth one located at the $K\alpha_{12}$ location. Additional refined parameters included the Soller slit angles with the “full” axial divergence model [24]. This analysis provided parameters describing the incident beam spectrum and Soller slit angles characterizing the IPF of the instrument utilizing the IBM. A second FPA analysis of SRM 660b, using data collected as per SRM 656, was performed; this provided final check on IPF parameters, such as the equatorial angle of the incident slit. With analyses of SRM 656, these IPF specific values were held fixed at the predetermined values. The refined lattice parameters from the five phase pure specimens were adjusted using the coefficient of thermal expansion found in Henderson and Taylor [25] to values in correspondence with 22.5°C .

The measurands are the certified values for lattice parameters obtained via the FPA with a Rietveld analysis are shown in Table 2. The statistical, Type A, evaluation of the lattice parameters resulted in estimates of the lattice parameter with $k = 2$ expanded uncertainties shown in Table 2. However, the systematic components of an additional uncertainty that were evaluated by Type B methods must also be considered. Data were considered primarily in the context of the uniformity in lattice parameter as a function of 2θ angle [26]; this, in turn, would reflect the functionality of the FPA model. This approach was applied to data from SRM 660b used to calibrate the machine. Another factor concerns the large amount of peak overlap in these two-phase mixtures; this leads to additional uncertainties in the ability to measure lattice parameters. Lastly is the concern of the small quantity of the β phase in the α 656, and likewise, the small quantity of α phase in the β 656, which renders the lattice parameters more difficult to determine in these phases. These considerations lead to an assignment of a Type B uncertainty of 40 fm for lattice parameters of the major phases and 50 fm for the minor ones. These considerations lead to the assignment of the Type A + B uncertainties shown in Table 3.

Table 2. Lattice Parameters Values and Type A Uncertainties for SRM 656 Powders

Material	Phase	Lattice Parameter a (nm)	Type A Uncertainty ($k = 2$)	Lattice Parameter c (nm)	Type A Uncertainty ($k = 2$)
α 656	α	0.775 411 05	0.000 016 56	0.562 034 40	0.000 009 71
	β	0.760 633 23	0.000 026 79	0.290 777 98	0.000 026 52
β 656	α	0.775 243 27	0.000 016 09	0.561 951 03	0.000 005 11
	β	0.760 363 03	0.000 002 37	0.290 742 47	0.000 004 74

Table 3. Certified Lattice Parameters of SRM 656

Material	Phase	Lattice Parameter a (nm)	Uncertainty Type A + B ($k = 2$)	Lattice Parameter c (nm)	Uncertainty Type A + B ($k = 2$)
α 656	α	0.775 411	0.000 043	0.562 034	0.000 041
	β	0.760 633	0.000 056	0.290 778	0.000 057
β 656	α	0.775 243	0.000 052	0.561 951	0.000 050
	β	0.760 363	0.000 040	0.290 742	0.000 040

(1)*: None detected.

Table 4. Information Microstructural Data for SRM 656

Material	Phase	Crystallite Size, Mass Percent Less Than (nm)					Microstrain, (ϵ_0) ⁽¹⁾
		10 %	25 %	50 %	75 %	90 %	
α 656	α	15.9	22.7	33.8	50.4	72.1	0.00024
	β	25.4	31.7	40.6	51.9	64.8	0.00048
β 656	α	13.3	20.7	33.8	55.3	86.0	*
	β	18.8	30.5	52.4	89.9	146.2	0.00044

For homogeneity verification, X-ray diffraction data were collected from two specimens from each of ten aforementioned bottles of the SRM materials. Specimens were prepared as 50/50 mixtures of SRMs 656 and 676. XRPD data were collected on a Siemens D500 diffractometer equipped with a focusing Ge incident beam monochromator, sample spinner and a scanning (proportional) position sensitive detector. Copper $K\alpha_1$ radiation was used. The scan range was from $17.5^\circ 2\theta$ to $152^\circ 2\theta$. GSAS was used for a Rietveld refinement of the three phases. Refined parameters included: scale factors, crystallite size and strain induced broadening terms of the pseudo-Voigt profile function [27], lattice parameters, sample shift and transparency terms, atomic thermal and position parameters for the silicon, aluminum and oxygen atoms, Lorentz polarization factor and six terms of a background function describing the effects of thermal diffuse scattering and air scattering (incoherent) of the incident beam. IPF specific values for the Gaussian U, V and W terms [28] and Lx and Ly were determined via a refinement of SRM 660 [29] and used as “floors” in subsequent refinements as described by Cline [15].

The relative intensities of the high intensity lines from the two SRM powders were determined from data collected on suites of five samples of the pure SRM materials. The RIR of the two polymorphs of silicon nitride (SRM 656) was determined from a suite of five samples consisting of 50-50 mixtures of the two SRM materials. Data from these specimens were collected on a Siemens D5000 diffractometer equipped with a sample spinner, graphite post monochromator and scintillation detector. Copper $K\alpha$ radiation was used. The divergence slit was set at 0.75° while a 0.2 mm receiving slit was used. The goniometer radius was 215 mm. Profile fitting was performed using the FPA method via TOPAS. Data from SRM 660a [30] were analyzed for determination of IPF parameters [15]. This analysis included the Cu $K\alpha_1/K\alpha_2$ emission spectrum with the satellite component and the “tube tails” [31,32]. With the refinement of the relative intensities, refined parameters included only the intensities and positions as well as the Gaussian and Lorentzian contributions to the sample induced broadening. The relative intensity data include the “relative intensity” of the peaks from the minor phase as well as the major phase from each powder. The scaling of these intensities is against the 100 % line of the major phase. The RIR data was biased using the certified quantitative data to result in a value which corresponded to a true 50:50 mixture of the α and β phases.

Table 5. Information Relative Intensity Data from the Two Silicon Nitride Powders of SRM 656

Phase & hkl		Two-theta	Relative Intensity (α 656)	Uncertainty	Relative Intensity (β 656)	Uncertainty
α	β					
101		20.59	100.00		12.85	± 0.27
110		22.91	45.79	± 0.34	5.79	± 0.06
	110	23.38	1.22	± 0.07	36.26	± 0.70
200		26.52	32.28	± 0.31	4.41	± 0.10
	020	27.05	4.43	± 0.07	100	
201		30.99	98.20	± 0.30	12.53	± 0.19
002		31.82	9.02	± 0.13	1.08	± 0.04
	011	33.67	4.91	± 0.09	91.24	± 0.62
102		34.55	85.05	± 0.51	10.66	± 0.11
210		35.34	94.53	± 0.36	12.60	± 0.12
	120	36.04	3.94	± 0.11	96.62	± 0.47

The Reference Intensity Ratio, $RIR_{\alpha/\beta}$, of SRM 656 is 0.719 ± 0.015 .

Information Values and Uncertainties: NIST information values are considered to be of interest to the SRM user, but are not certified because the measurements are not traceable to the SI, or only a limited number of analyses were performed which disallowed imparting plausible uncertainties to the measured values. The information values for the microstructural data, relative intensities and the RIR value, as determined from the analyses of the XRPD data, are given in Table 4 and 5, respectively. The particle size distribution, as determined by laser scattering, are given in Table 6. The interval defined by a value and its uncertainty is expressed as a $k = 2$ expanded uncertainties for the true value of the mean in the absence of systematic error. Additional data on elemental and chemical impurities of this SRM have been published in reference 33.

The hexagonal crystallite shape model used for analysis of the β phase of high β phase powder of SRM 656 included a shape parameter to model the observed anisotropic line broadening with a plate-like crystallite shape. The aspect ratio, the ratio of the edge length to the c axis dimension, refined to an average value of 5.5, indicating the strong plate-like crystal morphology. The dimensions shown in Table 4 are the edge dimensions. The presence of a plate-like crystal morphology of this phase was confirmed with a Scanning Electron Microscope examination. The anisotropic

broadening, however, could not be entirely accounted for the crystallite shape model. The additional broadening is due to the effects of planar faults perpendicular to the c axis. The presence of these faults has been confirmed by Transmission Electron Microscopy. Both the crystallite shape and stacking fault broadening affect reflections from planes intersecting the c axis; prismatic reflections are largely unaffected. One notes that the profile from 011 reflection of the β phase, listed in Table 5, displays said anisotropic broadening relative to the other lines listed.

The Rietveld analyses of the TOF and XRPD yielded structural data that did not differ significantly from the models reported by Marchand or Billy. However, analyses of the β phase using the XRPD data with either the structure proposed by Billy or Grün [34] could not be differentiated via consideration of the residual error terms.

Table 6. Typical Particle Size Data by Laser Scattering Method

Particle Size (μm)	Cumulative Mass Fraction Finer Than	
	α 656	β 656
0.3	0.3 %	4.5 %
0.6	20.8 %	43.5 %
0.9	48.6 %	66.8 %
1.2	64.2 %	78.4 %
1.5	76.2 %	85.9 %
1.8	85.2 %	90.9 %
2.1	91.3 %	94.1 %
2.4	95.0 %	96.2 %
2.7	97.2 %	97.6 %
3.0	98.5 %	98.5 %
4.0	99.8 %	99.6 %
5.0	100.0 %	100.0 %

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Certificate Revision History: 15 November 2017 (Recertification of phase purity and phase composition; addition of certified lattice parameter values; updated information values in the certificate; editorial changes); 23 September 2016 (Editorial changes); 20 September 2005 (Change in the certified values for phase composition resulting from the improved characterization of phase purity as provided in SRM 676); 30 March 1995 (Original certificate date).

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