Diffraction Metrology and Standards

Objective

Our objective is the development of Standard Reference Materials (SRMs) and quantitative, reproducible, and accurate measurement methods for characterization of any structure possessing spatial order on the scale of X-ray wavelengths. Such SRMs and measurement methods will aid in the development of new crystalline materials and devices made from them. Therefore, the impact of this project includes pharmaceuticals, ceramics, metals, semiconductors, and polymers.

Impact and Customers

- Our primary impact is the enhancement of the measurement capability of the laboratory X-ray diffractometer; there are 20,000 in use worldwide.
- Two of the three major instrument vendors supply NIST Diffraction Metrology SRMs with the equipment they sell. One vendor includes SRM 1976 with every instrument and, as a result, has been able to improve their angular resolution three times over the previous value.
- Calibration of equipment with NIST Diffraction Metrology SRMs is requisite for ISO certification.
- Primary customers are the International Center for Diffraction Data (ICDD) and X-ray instrument vendors (BEDE, Bruker AXS, Jordan Valley, PANalytical, Rigaku and Technos).

SRM	Material / Format	Diffraction Application	Unit Size (g)
640c	Silicon Powder	Line Position & Line Profile	7.5
675	Mica Powder	Line Position, Low 2θ	7.5
2000	Silicon 100 Wafer with Si/Ge epilayer	Hi-Resolution Line Position & Reflectometry	2.5 cm sq
660a	LaB_6 Powder	Line Position & Line Profile	6
1979	CeO_2 & ZnO Powders	Line Profile	New, In Prep
1976a	Sintered Alumina Plate	Instrument Response	2.6 cm disc x 0.2 cm
676a	Alumina (corundum) Powder	Quantitative Analysis	20
674b	Powder Set: ZnO, TiO_2, CeO_2, & Cr_2O_3	Quantitative Analysis	10 (each)
1878a	Respirable Quartz Powder	Quantitative Analysis	5
1879a	Respirable Cristobalite Powder	Quantitative Analysis	5
656	Silicon Nitride: a & ß Powders	Quantitative Analysis	10 (each)

Approach

Diffraction techniques can provide data on a number of sample characteristics. Therefore, the method of certification and the artifact itself are chosen to address a specific measurement issue pertinent to a diffraction experiment. NIST diffraction SRMs may be divided into five groups: Line Position, for calibration of the angle two-theta; Line Profile, for microstructure analysis; Instrument Response, for calibration of angle and intensity; Quantitative Analysis, for measurement of phase abundance; and Thin Film SRMs for measurement of thickness, surface roughness, and density of layered structures. The most common use of NIST SRMs is for calibration of diffraction line position. This requires that the SRM is certified with respect to lattice parameter. This length must be measured in a manner that is traceable to the International System of Units (SI) meter through a robust and transparent measurement chain. A major component of our effort consists of developing the capability for performing traceable measurement of lattice parameters for thin films and powders.

CERAMICS

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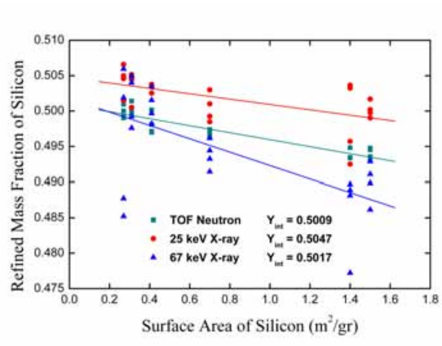
Accomplishments

The measurement technology associated with the certification procedures for the entire suite of diffraction metrology SRMs is continuously being improved. Three SRMs were recently certified, and a fourth is due for release early in 2009. SRM 676a is the renewal of SRM 676, an alumina powder certified for quantitative analysis of multiphase mixtures. Improvements in SRM 676a include an alumina powder (feedstock) of higher phase-purity, as well as a more accurate certification of its phase content. The new SRM 1976a is the successor to SRM 1976, which, since its introduction in 1991, has become the best selling of the diffraction metrology SRMs. SRM 1976a features a more uniform microstructure that, in turn, results in reduced error bounds on the certified values. The new SRM 2000 is the first thin-film, high-resolution diffraction SRM. It consists of 25 mm x 25 mm pieces of a silicon wafer with a Si-Ge epilayer. The SRM is certified with respect to the lattice parameter of the Si substrate. SRM 640d is also a renewal; SRM 640, silicon powder, was the first NIST SRM for powder diffraction and has traditionally been very popular. One of the leading instrument companies uses this SRM for ISO certification of equipment used in the pharmaceutical industry.



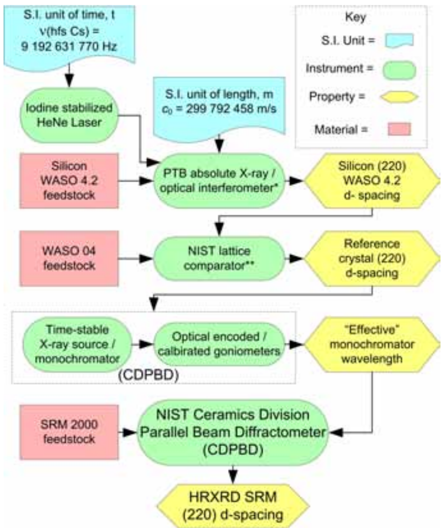
Refined diffraction data for SRM 676a

While diffraction is commonly used for quantification of multiphase mixtures, using it to quantify the amorphous-component fraction of unknown samples has only become possible with the certification of NIST SRMs for absolute phase purity. NIST's method for certification of phase content has been developed over the course of ten years and uses both synchrotron and neutron powder diffraction methods. The method's first application was in the recertification of SRM 676. With SRM 676a, further improvements to the experimental design realized the convergence of three measurement methods (neutron time-of-flight scattering and X-ray diffraction on two different synchrotron beamlines, at energies of 25 and 67 KeV); this allowed certification of SRM 676a with reduced error bounds. SRM 676a will also enable accurate measurement of the thickness of disordered surface layers, which is of particular importance for nanoscale materials with surface-dependent properties.

SRM 1976 was originally certified in response to a round robin study done by the International Centre for Diffraction Data in preparation for a new database format. A conclusion of the study was that an SRM was needed that allowed for the calibration of both line position and intensity as a function of two-theta angle (instrument response) using conventional data analysis methods. Based upon customer feedback on SRM 1976, the new SRM 1976a was manufactured in a single production run in order to significantly reduce the error bounds in the relative intensity data.

SRM 2000 provides the high-resolution X-ray diffraction (HRXRD) community with either wavelength or angle calibration through certification of the Si (220) lattice spacing of the artifact. This SRM also provides certification of the Si wafer miscut (angle from surface to crystal plane) for sample rotation axis and sample holder alignment. Future recertification of this SRM will involve modeling HRXRD reflection data to provide customers with epitaxial layer structural information.

The silicon used for SRM 640d was obtained from intrinsic, float-zone boules that were crushed and jet-milled to a narrow particle size distribution centered at 5 micrometers. The powder was than annealed to remove crystallographic defects caused by comminution. This preparation procedure yields a powder that is optimal for powder diffraction measurements. The certification measurements were performed on the newly commissioned Ceramics Division Divergent Beam Diffractometer (CDDBD).



SI traceability chart for SRM 2000

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Publications

SRM 676a; *Alumina Powder for Quantitative Analysis by X-ray Powder Diffraction*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (2008)

SRM 1976a; *Instrument Response Standard for X-ray Powder*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (2008)

SRM 2000; *Calibration Standard for High-Resolution X-ray Diffraction*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (2008)