Diffraction Metrology and Standards

Both powder and high-resolution diffraction can provide a wealth of information with a relatively simple scan. However, the data are beset by a complex error function. It is the characterization of this function, critical to the accuracy of the technique, that is one of the prime functions of NIST diffraction Standard Reference Materials (SRMs). In order to be traceable to Systeme Internationale (SI) based units, certification measurements must be performed on equipment that features goniometers that are self-calibrating, i.e., equipped with stable, high-resolution encoders that have been calibrated using the circle closure method.

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This project establishes the traceability of NIST diffraction SRMs certified with respect to lattice parameter or layer spacing. From Bragg's Law, $\lambda = 2d \sin\theta$, we see that traceability in spacing, d, requires both traceabilty in wavelength, λ, and angle, θ. The linkage of wavelength to the SI has been developed, and follows the first four levels in the traceability diagram shown in Figure 1. Here we devise a rigorous approach for the calibration of the goniometers in the Ceramics Division parallel beam diffractometer, establishing the traceabilty of angle measurement in future SRM certification.

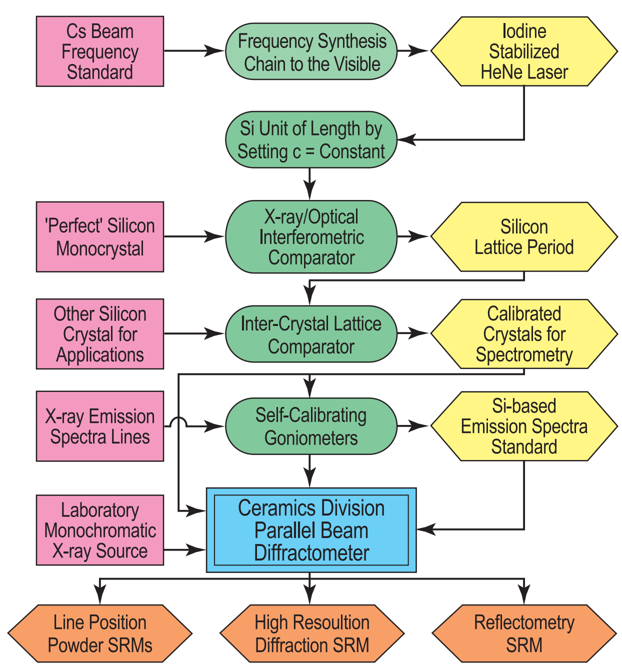

Figure 1: *Measurement chain of NIST certified diffraction SRMs.*

To be useful in a metrological context, a goniometer must have a stable, accurate and precise optical encoder. However, the encoder will exhibit an error function that must be characterized. The term "self-calibrating" refers to the ability of the goniometer to measure angles in a metrologically credible manner without relying on an external artifact and is realizable through the method of circle closure. A caveat to the method is that encoder errors must only be long range in nature, *i.e.*, caused by mounting misalignment, eccentricity, etc. In order to realize a conceptual understanding of the circle closure method, assume the goniometer of interest is equipped with a second, coaxially mounted rotation stage. This second stage rotates two optical faces, mounted parallel to the rotation axis and exhibiting a 30° dihedral angle. A scanning autocollimator is used to generate a feature associated with the angle of each face. The fitting of this feature results in a measurement precision of the angle between the faces that is much greater than that offered by the encoder itself. After each measurement, the second stage is rotated 30°, and the process repeated to yield a series of data points through 360°. Circle closure asserts that the sum of the angle measurements for one complete axis rotation must be 360°, and therefore, all errors sum to zero. Thus, the average of the measured dihedral angles provides the true angle, and deviations from this angle map the encoder correction curve.

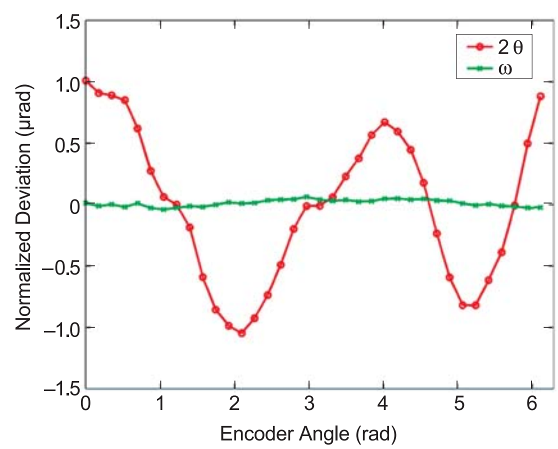

Figure 2: *Encoder error corrections from circle closure.*

We utilize a goniometer assembly with two axes of interest and a third stage for rotation of optical faces. This approach provides a matrix of encoder errors. Matrix inversion yields the calibration curves, shown in Figure 2, for the ω and 2θ goniometers.

Contributors and Collaborators

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