Published in final edited form as:

Acta Crystallogr A Found Adv. 2019 January 01; 75(Pt 1): 158–164. doi:10.1107/S2053273318016935.

Model-independent extraction of the shapes and Fourier transforms from patterns of partially-overlapped peaks with extended tails

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Abstract

This work presents a technique for extracting the detailed shape of peaks with extended, overlapping tails in an X-ray powder diffraction pattern. The application we discuss concerns crystallite size broadening, though the technique can be applied to spectra of any origin and without regards to how the profiles are to be subsequently analyzed. Historically, the extraction of profile shapes has been difficult due to the complexity of determining the background under the peak, resulting in an offset of the low-frequency components of the Fourier transform of the peak known as the “hook” problem. The use of a carefully considered statistical weighting function in a non-linear least-squares fit, followed by summing the residuals from such a fit with the fit itself, allows one to extract the full shape of an isolated peak, without contributions from either the background or adjacent peaks. The extracted shape, consisting of the fit function recombined with the residuals, is not dependent on any specific shape model. The application of this to analysis of microstructure is performed independently of global parametric models, which would reduce the number of refined parameters; therefore the technique requires high-quality data to produce results of interest. The effectiveness of the technique is demonstrated by extraction of Fourier transforms of peaks from two sets of size-broadened materials with two differing pieces of equipment.

1. Introduction

It is well known that the profiles of an X-ray powder diffraction (XRPD) pattern contain information about the microstructure of the sample. Bertaut (Bertaut, 1950) presents an *ab initio* derivation of the relationship between the length distribution of coherent columns of a crystallite, as observed perpendicular to the diffraction direction, and the Fourier transform of the specimen diffraction profile. Direct application of this technique, however, has always been problematic. The vast majority of data analysis techniques rely on non-linear least-squares fitting of model functions onto the observation. This approach allows for a substantial reduction in the number of fitting parameters from what would be required to analyze each peak independently in Fourier space. However, the result is difficult to connect back to the underlying Bertaut theory, since it is then dependent upon the models chosen, and upon how errors in such models affect the column length distributions.

The validity of the Bertaut theory is dependent upon correct extraction of the full Fourier spectrum from the diffraction profile. With the powder diffraction experiment, this condition manifests itself in the determination of the correct background level and the shape of a function which may extend very far from the center of a reflection. The difficulty is exacerbated by the shapes of XRPD peaks; such peaks typically have Lorentzian tails, which are asymptotically proportional to $1/(x - x_0)^2$, where x is a diffraction angle and x_0 is the peak center. This type of tail extends out to very long distances from the center, and often encroaches on neighboring peaks. Also, the peak shape itself can be quite complex, due to the convolved contributions of the column length distributions and the instrument profile function (IPF). Thus, without relying on a detailed model (and reintroducing unknown model dependencies) it is quite difficult to derive the shape of an isolated peak, as required for Fourier analysis and the application of Bertaut theory.

An illustration of this difficulty is shown in figure 1. The curves show the discrete Fourier transform of a Lorentzian function

$$y(x) = \frac{a/\pi}{a^2 + x^2}$$

with $a = 1$, but with the tails of the peak truncated at various distances from its center and shifted vertically to put the tail at $y = 0$. The inset shows the actual peaks. This is very similar to what happens with real baseline subtraction of a peak. Note that these transforms are plotted without renormalizing the amplitude to unity at the origin to emphasize the source of the problem being reduced low-frequency content relative to the high-frequency regions. The exact Fourier transform of the non-truncated function $y(x)$ is $\mathcal{F}(\omega) = e^{-|a\omega|}$ (the dashed, orange curve) where ω is the transform variable conjugate to x . Even with the peak tails extended out to 10 full widths at half maximum (FWHM) on each side, the “hook”, and associated low slope is very evident. Note that all the curves converge to the same value at higher frequencies. Also note that, as the peak tails get truncated, there are fewer and fewer points available for Fourier transform, so resolving the slope at the origin becomes progressively more difficult. Since the slope of the transform of a peak at $\omega = 0$ is related inversely to the area-weighted mean column length $\langle L \rangle_A$ (Bertaut, 1950), the result can be very far off, resulting in completely unphysical results. The problem, discussed in detail in (Delhez *et al.*, 1988) is nicely summarized in (Delhez *et al.*, 1980): “Estimation of the background level is still more an art than a science”. In the past the consequences of truncation were mainly addressed by adding profile tails estimated from the central part of the profile recorded, *e.g.* (Vermeulen *et al.*, 1991). These references, however, address a situation in which insufficient background information is available to fit the entire peak and background together; this is not applicable to our situation. Here we present a technique that estimates the profile tails from the diffraction angle regions where the profiles overlap, *i.e.*, excluding the peak centers.

This work presents a new technique that allows one to correctly isolate a peak over the tails of nearby peaks and a smooth background. The data are fit in a two step process. In the first fit, a simple analytical profile shape function (PSF), typically a Voigt function, is used with

the weighting function being adjusted so as to favor the accurate fit in the tail region of the profile. The second step recombines the localized residuals from the first fit with the fitted PSF itself. By separating the data this way, one can determine quite accurately the structure of the peak above the local background and the tails of nearby peaks. In the first step, the statistical weighting w used to compute χ^2 from the computed y_{calc} and observed y_{obs} values,

$$\chi^2 = \sum w(y_{\text{calc}} - y_{\text{obs}})^2 \quad (1)$$

which is minimized by least-squares fitting, may be altered from the usual $w = 1/\sigma^2$ (where σ^2 is the variance associated with each datum), the goal being to favor the correct fitting of the tails, at the cost of fidelity at the center of the peak or statistical correctness. The resulting fit is an accurate representation of the smooth parts of the data set between the peaks at the expense of any interest in accuracy near the center of the peak. Since the tails were fit with care, the residuals are highly localized around the center of the peak, and it is straightforward to identify a region around the peak center in which the residuals are non-zero. The recombination of the localized residuals with a fit that encompasses the long-range tails allows one to reconstruct the shape of a peak without limiting the result to one that can be represented by a closed-form PSF. Extraction of the profile in full detail is critical as crystallites have shapes and size distributions that create column-length distributions that, in combination with the IPF, result in peaks of complex shapes in both real space and Fourier space.

As presented, the technique is appropriate for low-noise, high-quality data from nearly monochromatic sources. We demonstrate it with data sets taken at a synchrotron and with a laboratory diffractometer equipped with a Johansson incident beam monochromator (IBM) and position sensitive detector (which provides very high counting statistics). Without an IBM, attempts to extract the Fourier transforms of peaks will typically be confounded by ripples in the transforms of the IPF introduced by interferences between multiple near-by lines (*e.g.* $KL_3(Ka_1)$ and $KL_2(Ka_2)$) in the x-ray spectrum. The technique can be applied in the case of peaks that are at least partly resolved (having a significant dip between the maxima) and sitting on a background that is known to be free of features that could be confused in a fitting procedure with the tails of the peaks. The technique addresses two issues in the analysis of a diffraction pattern: first, it mitigates the effect of the overlap of tails on peaks and, second, it allows the direct extraction of the Fourier transform of an arbitrarily-shaped peak. This latter facet is critical to a microstructure analysis as it has been demonstrated that broadening due to such features does not result in profiles describable with analytical PSFs (Scardi & Leoni, 2001; Ungár *et al.*, 2001). Although throughout this work, we will refer to Lorentzian tails, since they are the most common tail shape observed in XRPD. The method we will describe can be applied to any function for which the asymptotic shape of the tail is well known, and is not restricted to situations for which Fourier analysis is the goal. Many realistic profiles are actually of the “super”-Lorentzian form (a sum of Lorentzians of differing widths). The asymptotic behavior of such a peak is Lorentzian, with corrections of order $1/x^4$ and higher powers closer to the center of the peak,

where the residuals will be explicitly included. These higher powers are equivalent to power-series expansions such as those of (Vermeulen *et al.*, 1991). Such peak shapes are well-handled by this technique. The interpretation of the extracted shape from each individual peak is up to the user of this technique; application of specific models such as crystallite size, shape and microstrain, which result in systematic variation of peak shapes based on diffraction angle and symmetry properties of a reflection, is not within the scope of this paper.

2. Profile Fitting and extraction of transforms

We consider the issue of how data are weighted in a least-squares refinement. For normal Poisson-statistics, one weights the data with

$$w = 1/\sigma^2 = 1/(N + 1) \quad (2)$$

where N is the number of counts in a bin of the pattern (Kirkpatrick & Young, 2009). For some data sets, this weighting works well for the purposes described below. In other cases, though, a modified weighting can be applied. The modified weighting is

$$w = 1/(1 + N + a^2 N^2) \quad (3)$$

and, typically, $a \approx 0.1$, places more emphasis on the bins with small numbers of counts, and less on the peaks. The actual value of a can be tuned for specific cases. Other weighting functions can be used if appropriate to produce better behavior of the function tails; the choices presented above work for the data sets we will present. The effect is that the fitted function agrees very well with the tail regions of the peaks, and much less well with the center.

However, since the tails are fit very well, the residuals from the fit are very localized around the peaks, and can then be isolated on a peak-by-peak basis for further analysis. The choice among fit functions and weighting schemes must be made by examining the residuals of sample fits; whichever peak shape and weighting causes the residuals to return to zero most rapidly is likely to be the better choice here. It is important to note that the final peak shapes determined from the procedure are nearly independent of this tuning; as long as the fit regions around the peaks include the entire region of non-null residuals, the outcome is not affected. It is this property of the technique which makes it model-independent, since errors in the peak model are swept into the residuals, and then re-incorporated in to the final analysis. However, optimizing the weights at this point will have the effect, by finding a choice which makes the residuals very localized, of reducing the uncertainties in the fits, since poorly-localized residuals require analysis over a wider region, thus incorporating more noise. The only restriction on the function chosen for the analytic peak shape is that its Fourier transform be calculable, preferably in closed form. In the examples below, we work with symmetric functions, but this is not a requirement. However, in many cases, far from the center, the symmetric component of Lorentzian tails dominate the peak, and the asymmetry can be absorbed in the residuals, so that even with modestly asymmetric measured peaks, a symmetric fit function suffices.

We first consider data collected at the Advanced Photon Source (APS) on the 11BM high-resolution powder diffractometer (Wang *et al.*, 2008); the IPF of this machine is such that data can be readily fit with a symmetric Voigt PSF. Lorentzian profiles were also tested using the two step process, and produced essentially identical results; the residuals in each case are accounted for exactly in the second stage of the analysis. However, since the initial Voigt fit produced much smaller residuals, it is considered to be a better starting point for the analysis.

The Voigt function, in the form we express it, has two parameters, the half-width at half-maximum (HWHM) of the Lorentzian, w , and the standard deviation of the Gaussian σ . The functional form, then, is

$$V(w, \sigma; x) = \frac{1}{x^2 + w^2} \otimes e^{-\frac{x^2}{2\sigma^2}}, \quad (4)$$

where $\otimes$ is the convolution operator. For our purposes, this is generated numerically by the algorithm of (Mendenhall, 2007). Also, note that the exact Fourier transform of the Voigt is

$$\mathcal{F}(V)(w, \sigma; \omega) = \exp\left[-w\left|\omega\right| - \frac{\sigma^2\omega^2}{2}\right]. \quad (5)$$

Using these Voigt functions, one for each reflection, the weighting for Poisson-statistics described above, and a background consisting of (typically) Chebyshev polynomials in 2θ , we carry out a nonlinear least-squares (NLLSQ) fit to the entire pattern to compute a good approximation to the shape of the pattern everywhere except on top of the peaks. The next step is to compute the residual set $R(x_j)$ of the resulting fit,

$$R(x_j) = y_j - bg(x_j) - \sum_m C_m V(w_m, \sigma_m; x_j - X_m), \quad (6)$$

where x_j is the j^{th} abscissa, $bg(x_j)$ is the background value calculated from the fit at x_j , and C_m , w_m , σ_m , and X_m are the area, Lorentzian width, Gaussian σ and center, respectively, of the m^{th} fit peak. It is very important that the background function chosen be capable of describing the real background in the system, without having so many free parameters as to allow it to start to mimic the tails of peaks. Note that using a global fit encompassing all the peaks at this point strongly constrains the background function, so it cannot adjust itself to shift the baseline under individual peaks, which would be the source of a “hook”.

Next, the pattern is divided into regions around each peak where the residuals are non-zero. Because of the short-range tails of the residuals, it is usually unambiguous where one can put a cutoff for each peak such that all the residual information is encompassed in each region without it colliding with the residuals from any adjacent peaks. We use an adaptive procedure that creates a window width around peak k that is

$$W_m = r\text{FWHM}_m \sqrt{\frac{C_m}{\text{FWHM}_m} / \text{bg}(X_m)}, \quad (7)$$

where r is typically about 1. This is just the FWHM of a peak scaled by a multiple of its signal/background ratio. If this window encroaches on the window of another peak on either side, it is clipped to the halfway point between the peaks on that side. This procedure is empirical, but seems to generate windows that correspond well with the detectably non-zero region of the residuals. Depending on the particular characteristics of a data set, one may tune r to encompass the residuals better, or may entirely hand-select the peak windows if this procedure does not perform well.

The case in which classical Poisson weighting (eq. 2) works well is shown figure 2 which displays fits of the Voigt PSF to data from the “15nm” crystallite size of the ZnO material of NIST Standard Reference Material (SRM) 1979 (NIST, 2016) collected on 11BM. Given the high statistical quality of these data, and that the profiles are inherently “Voigtian” we expect the Poisson-statistics will provide a quality fit to tails of the profiles. In contrast to this, wherein it is appropriate to use the relative-error weighting (eq. 3), are the fits shown in figure 3. These are fits to data from the “60nm” crystallite size SRM 1979 collected on the NIST Divergent Beam Diffractometer (DBD) set up with a Johansson IBM (Cline *et al.*, 2015). The IPF of this machine is quite complex. In figure 2 the blue difference curve, corresponding to Poisson weighting, shows a sharp return to baseline away from the center of the peaks. The difference curve shown in red, however, corresponding to use of modified weighting (eq. 3), illustrates that the return to background is less clear. For the Poisson weighting, it is very easy to distinguish a region in which the residuals are non-zero from a region in which they vanish, and analyze that region in combination with the analytical fit to reconstruct the full shape of the peak. Even for the relatively wide and overlapped peaks in the region between $d^* = 7 \text{ nm}^{-1}$ and $d^* = 7.5 \text{ nm}^{-1}$, the separation is complete. Figure 3 shows the reversed case, where modified (red difference curve) weighting provides much better suppression of the tails. This may be the case with wide or asymmetric peaks. The choice should be made by examining test fits.

We then compute, by weighted least squares, the Fourier transform of the residuals associated with each peak. Note that, since the transform is computed by least squares, we are incorporating correct statistical information from the original counting statistics. The errors in the residuals are perfectly anti-correlated with errors in the fit, since the residuals are just $\text{data} - \text{fit}$. Since the final result will be the sum of the analytic transform of the Voigt functions, plus the transform of the residuals, we can treat the fit as having no uncertainty, and assign the original $\sigma^2 = n + 1$ statistics, from a bin containing n counts, to a residual bin. This resulting Fourier transform can then be summed with the Voigt transform from eq. 5 (or transform of any other analytic fit function which serves the purpose of creating localized residuals) to produce a final transform associated with a specific reflection. Thus, for the Voigt fit, the shape of a peak is represented as:

$$f_m(x) = C_m W_m V(w_m, \sigma_m; x - X_m) + A_0 + \sum_{k=1}^n A_{mk} \cos k\omega_0(x - X_m) \quad (8)$$

$$+ \sum_{k=1}^n B_{mk} \sin k\omega_0(x - X_m),$$

where the A_{mk} and B_{mk} come from the least squares fit to the residuals in the window for peak m , zero-padded outside the window to allow the Fourier transform to be computed at low frequency, and the factor W_m is to normalize the continuous transform of the Voigt function to a discrete transform. The Fourier base frequency $\omega_0 = 2\pi/x$ where x is the full width of the region over which the residuals were analyzed, including any zero-padding, to compute the A and B coefficients, in 2θ space. Critically, if the fit is carried out by properly-weighted least-squares, these coefficients also have uncertainties and a full variance–covariance matrix attached to them that can be used in the subsequent interpretation of the transforms. This is in sharp contrast to the commonly used Fast Fourier Transform (FFT) technique wherein said errors are not propagated. An FFT is a least-squares solution to a problem of uniformly-sampled, uniformly-weighted data, not a full, general problem. The discrete transform is then:

$$\mathcal{F}(f)(k\omega_0) = C \exp \left[-w|k\omega_0| - \frac{k^2 \sigma^2 \omega_0^2}{2} \right] + A_k + iB_k. \quad (9)$$

For data sets for which the peak isolation of the previous discussion has worked correctly, the residuals are completely non-overlapping, allowing assignment of the Fourier transform entirely to a specific peak, with no mixing of coefficients. Also, since the residuals are small at the boundary of the window over which they are being summed, any discontinuity introduced by assuming a zero value for them outside the window is minimal. The raw Fourier transforms thus produced contain both the required information about the material, and the IPF, referred to as g_{IPF} , that is to be removed. Since the material peak shape and g_{IPF} are combined by convolution, in the Fourier domain, the transforms are multiplied. To remove the effects of g_{IPF} , we divide by its transform $\mathcal{F}(g_{\text{IPF}})$, as computed from a Fundamental Parameters Approach (FPA) (Cheary & Coelho, 1992) peak model for the machine. This transform can then be interpolated onto the same ω grid as used in eq. 9, and the division carried out. Thus, if $\mathcal{F}(g_{\text{IPF}})(k\omega_0)$ is the transform of the IPF,

$$\mathcal{F}(f_1)(k\omega_0) = \frac{C \exp \left[-w|k\omega_0| - \frac{k^2 \sigma^2 \omega_0^2}{2} \right] + A_k + iB_k}{\mathcal{F}(g_{\text{IPF}})(k\omega_0)} \quad (10)$$

is the transform of the intrinsic peak shape due to the material being analyzed. This is essentially the Stokes method (Stokes, 1948) of deconvolution. The uncertainties of the A_k

and B_k from the least-squares fit variance-covariance matrix are similarly scaled by $1/\mathcal{F}(g_{\text{IPF}})$ and carried forwards. This is critical to further analysis, and differentiates this technique from many older works (Zorn, 1988); however, handling of uncertainties for specific conditions has been presented in §3.3 of (Delhez *et al.*, 1980), based on the papers of Wilson (Wilson, 1967; Wilson, 1968; Wilson, 1969). The real part of this transform is what appears in the Bertaut equations. The transform for a symmetrical, centered peak is purely real; to adjust for centering, we multiply this transform by $\exp - ik\omega_0$ with selected to get a slowly varying phase as a function of k .

It is worth noting that, once the residuals for a peak from equations 6 and 7 have been constructed, it would be equally possible to apply a stabilized direct-space parametric peak shape fit to the sum of the Voigt and the residuals, in the manner of (Louër *et al.*, 1969). However, since the most likely application of this technique is to compute the Fourier transform of the peak, to be applied to the Bertaut theory, we do not do this.

3. Example Fourier transform results

We demonstrate the efficacy of the technique with an analysis of crystallite size broadening. Data were collected on materials of differing crystallite size distributions and two machines of differing resolution. This presents a situation wherein the analysis is based entirely on the Fourier transforms of the peak shapes, and will only yield consistent results if peak shapes are correctly extracted with a particular sensitivity to the background issue. Two of the samples were the “15nm” and “60nm” SRM 1979 powders discussed above, along with Al_2O_3 (corundum structure) powder of SRM 676a (NIST, 2008). The real part of a subset of the Fourier transforms resulting from the analysis for the high-resolution “15nm” SRM 1979 data are shown in figure 4. This material has a strong hk dependence of the peak widths due to stacking faults as per ref. (Langford *et al.*, 1993). The reflections shown in figure 4 that are not affected by stacking faults, “group 1”, are the 002 and 004 reflections. Two measurements for each reflection are shown as the solid curves; the consistency of these transforms, across multiple reflections, demonstrates the stability of this Fourier transform extraction method. The dashed lines represent two measurements of the 102 reflection, “group 3”, that exhibit the maximum degree of stacking fault broadening; we observed consistency between the two measurements.

We also assess the validity of the method by comparing the results from two machines of widely differing resolutions: the APS 11BM and the NIST DBD. Figure 5 shows the area-weighted and volume-weighted mean column lengths from both of the SRM 1979 materials, as described by Bertaut (Bertaut, 1950). Instrumental corrections were applied as per eq. 10. The data from the DBD were processed using the relative error weighing as per eq. 3 while the data from 11BM were processed using eq. 2. For the “15nm” particles, which have relatively broad lines that are entirely resolved with either the DBD or 11BM, the agreement between the data sets is complete. However, for the “60nm” crystallites, which are approaching the resolution limit for the DBD, one starts to see some discrepancies, especially at low angles where the axial divergence of the DBD broadens the IPF. This is particularly significant for the volume-weighted mean column length $\langle L \rangle_v$, which is sensitive to the high frequency components of the Fourier transforms, and thus sensitive to

the cutoff of IPF in Fourier space. The area weighted mean column length $\langle L \rangle_A$ only depends on the slope of the Fourier transform at $\omega = 0$, so is relatively insensitive to the IPF; however, this characteristic makes it very sensitive to the “hook” effect, which would flatten the curve at low frequency and typically result in an overestimate of $\langle L \rangle_A$.

The SRM 676a data has a higher noise floor, and we choose a region of fairly severe peak overlap to test this technique under more challenging conditions, approaching the limits of what this method can reliably achieve. Figure 6 shows a small region from the SRM 676a data. Note the peak pair at $d^* = 12.55\text{nm}^{-1}$ in figure 6 that is not resolved. The appropriate positions for the analysis windows, shown by the green bars, is unclear in this case. In that case, we put the boundary halfway between the positions of the two underlying peaks. Also, note that there are features in the data, appearing as shoulders just to the left of the 054 reflection and the 330 reflection; cutting off the analysis window close to the peak mitigates the contribution of these to the Fourier analysis, but there is no “right” way to handle these without understanding their source.

Figure 7 shows the computed Fourier transforms for the seven peaks in figure 6. The black curve corresponds to the very small peak on the left of figure 6; its result is quite limited by the noise floor. Even the orange curve, which corresponds to the badly overrun peak at $d^* = 12.555\text{nm}^{-1}$, gives a reasonably consistent transform. This is due to the fact that most of the shape of the transform comes from the Voigt fit, and little from the residuals. This material has very little strain or faulting, so it is expected that these transforms have approximately the same shape as shown in figure 7.

4. Conclusions

The long-standing difficulty with computing the Fourier transforms of peaks in X-ray powder diffraction patterns can be resolved by a fitting procedure that assures accurate representation of the tail shapes for peaks, combined with re-incorporation of the residuals from the fit into the data set to be transformed. This process cleanly separates the long-range and short-range components of the data, and addresses the “hook” problem. It enables computation of the real-space shape and Fourier transform of each peak in a powder pattern independently of other peaks. As a specific example of the application of this technique, we compute microstructural information derived from data from SRM 1979 collected on both the NIST DBD and APS 11BM machines. The results compared well, despite fundamental differences in their respective IPFs. Complete agreement was observed with the “15nm” material. With the “60nm” material, consistency was observed between the $\langle L \rangle_A$ values that are particularly sensitive to the “hook” effect, indicating that this issue has been resolved. Divergence was observed with respect to the $\langle L \rangle_V$ values as the resolution limits of the DBD were approached; though identical trends in the “60nm” data were nonetheless observed between the two instruments. Use of this technique to compute properties of peaks, independent of global structural models that constrain peak-shape parameters across a diffraction pattern, should be considered only for data sets of very high statistical quality.

Acknowledgment:

Use of the Advanced Photon Source at Argonne National Laboratory was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. Official contribution of the National Institute of Standards and Technology; not subject to copyright in the United States.

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Figure 1.

Illustration of "hook" effect in a Fourier transform of a Lorentzian peak due to truncation of peak tails. Legend text specifies the cutoff of the tails, in units of the FWHM. Inset illustrates cuts, with colors corresponding to colors of curves in main plot. Fourier transforms are not renormalized to have amplitude 1 at the origin.

Figure 2. Residual errors from fits of the Voigt PSF to the “15 nm” SRM 1979 data collected on APS 11BM. Residuals are from the use of both Poisson weighting (eq. 2) and relative-error weighting (eq. 3).

Figure 3.

Residual errors from fits of the Voigt PSF to the “60 nm” SRM 1979 data taken on the NIST DBD. Residuals are from the use of both Poisson weighting (eq. 2) and relative-error weighting (eq. 3).

Figure 4.

Fourier transforms of reflections from the “15nm” SRM 1979 material from the 11BM data. Horizontal scale is in nm. Dashed curves are samples from a reflection with stacking fault broadening. Solid curves are samples from multiple reflections which do not exhibit stacking fault broadening. Inset is same plot on log scale.

Figure 5.

Mean column lengths of SRM 1979 “15 nm” and “60 nm” materials, comparing values from the 11BM data to the those from the NIST DBD.

Figure 6.

Typical region around peaks from SRM 676a data taken at APS 11BM. Every fourth data point is plotted.

Figure 7.
Fourier transforms of individual reflections shown in figure 6 from the SRM 676a material.