

An Improved Workflow for Reproducible Processing and Analysis of Polycrystalline Electron Diffraction Patterns

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Successful quantitative analysis of electron diffraction patterns from thin, polycrystalline samples requires careful attention to detail at each stage. First, one must record patterns from the unknown and standard under equivalent electron optical conditions (equivalent lens currents corrected for hysteresis.) Next, one must locate the center of each pattern and measure and correct for the elliptical distortion [1, 2, 3] and compute a radial average. The continuous scattering (background) must be subtracted. Peak positions from the standard are then used to determine the camera constant and calibrate the unknown pattern. The peaks in the unknown may then be compared to those expected from candidate structures.

Until recently analysts in our laboratory have used a pair of programs with graphical user interfaces to locate the center and compute the elliptical distortion [2] and to correct the distortion, calibrate the pattern, and compare [3] to the ICDD diffraction database. This analysis required a lot of “point-click-copy-paste” by the analyst and was difficult to automate. This report summarizes the development of a more efficient and reproducible workflow. Our tool chain permits more automated report generation as outlined by Vandewalle *et al.*[4].

To obtain the required precision, the pattern from the standard is acquired after that of the unknown using the same lens currents, corrected for hysteresis by a normalization procedure in both the imaging and diffraction mode of the microscope. The analyst uses a DigitalMicrograph plug-in, EDP, derived from VHou’s plug-in [2] and extended to correct the distortion as outlined by Capitani *et al.*[1], perform the radial average, and output the profile as a comma-delimited file. All the postprocessing uses the Open Source R language. The core functions (unit operations) to tune the background subtraction of the radially-averaged intensity, measure the camera constant from the standard, plot a calibrated background-subtracted profile from an unknown pattern, and overlay lines from known phases were encapsulated in a custom R package, *edp*, which permits an analyst to tune and script an analysis of a new material with a minimum amount of coding. Background subtraction and peak location functions were used from the R package, *Peaks*[5]. This approach is consistent with the “do not repeat yourself” (DRY) coding practice to minimize inconsistencies. The *edp* package contains data and examples (unit tests) that are run each time the package is built and checked. The source code for both our DigitalMicrograph plug-in and the R package are maintained under version control using the distributed version control program, *git*. We intend to release both packages under the Gnu Public License.

A typical workflow is to tune the background subtraction for a new unknown pattern, measure the camera constant for the standard, display the pattern and compare to candidate phases using a script using the Open Source RStudio integrated development environment. The Sweave package in R permits the analyst to embed these “code chunks” and the report text (in $\text{L}^{\text{A}}\text{T}_{\text{E}}\text{X}$ format) into a file that is compiled by RStudio to produce a final report in PDF format. Judicious use of templates minimizes editing. Because the sources for these reports are text files, it is easy to maintain a project under version control. At the end of the project, the analyst simply compresses the entire folder with the report and all required supporting data to reproduce the analysis into a compendium with 7zip. After locating the compendium months later, the analyst can reproduce the analysis in less than 15 minutes. This approach is easily adapted to other analyses in a quantitative microscopy laboratory.

References

- [1] G Capitani et al., *Ultramicroscopy* 106 (2006)p. 66-74.
- [2] V Hou and D Li, *Microscopy Today* 16 (May2008) p. 36-41.
- [3] J L'ab'ar and M. Adamik, *Microscopy and Microanalysis* 7(Suppl 2) (2001)p. 372-373.
- [4] P Vandewalle et al., *IEEE Signal Processing Magazine* 26(3) (2009)p. 37-47.
- [5] M.Morhac, *PeaksPackage* v. 0.2, *ComprehensiveR Archive Network*(2012).

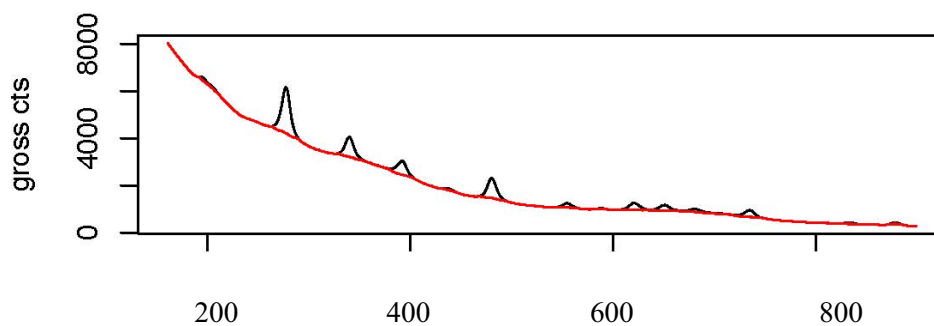


Figure 1. A radially-averaged electron diffraction pattern from a layer of BaTiO_3 nanoparticles and the calculated background. X-axis represents r [px]

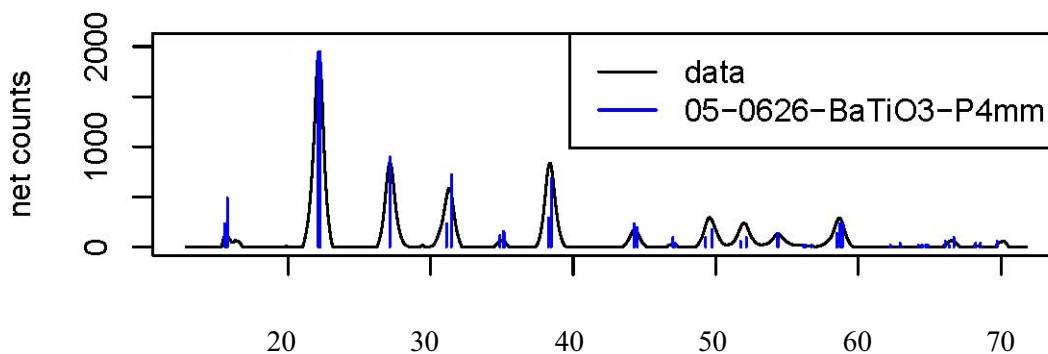


Figure 2. The calibrated, background-subtracted electron diffraction pattern from a layer of BaTiO_3 nanoparticles with the predicted lines from the ICDD database