A straightforward program written in Octave and Julia for X-ray diffraction line profile analysis: A study in gold thin films

Adriana Pérez-Casanova,[1] Héctor Medel-Cobaxin[1] and Flavio F. Contreras-Torres*[1,2] (contreras.flavio@itesm.mx)
[1]Escuela de Ingeniería y Ciencias, Tecnológico de Monterrey, Monterrey 64849, Mexico
[2] Centro del Agua para América Latina y el Caribe, Tecnológico de Monterrey, Monterrey 64849, Mexico

From the concentrations and distributions of crystal defects (e.g., dislocations, stacking and twin faults, lattice distortions, vacancies) to the crystallite size and shape distributions, and the amount and distribution of phases, the microstructural properties of polycrystalline materials can be analyzed through theoretical approaches well established and in state-steady level. Accordingly, the study of the microstructure underlay the use of routines specially developed for bulk materials (i.e. powders), which are classified on how they can analyze the diffraction-line broadening from experimental XRD data. However, such mathematical treatments with more than one evaluation strategy and several ways for its implementation cannot be properly suitable for the evaluation in nanocrystalline thin films[1]. In particular, negative microdistortions and coherently diffracting domain sizes have been calculated in platinium and gold thin films, while the original concept of strain and size only allows positive values[2]. In this concern, the smoothing routines, the procedures for $k\alpha_2$ elimination as well as the calculation of integral breadth values are very critical. Smoothing routines are used to reduce the random noise, while the $k\alpha_2$ elimination is needed to obtain proper values of integral breadths. Such tasks are usually implemented in commercial software packages, thus setting restrictions on modifications into the routines of the code.

In this study, we show the development of a computational program to implement strategies of Line Profile Analysis for crystalline materials. After considering the experimental setup for XRD data acquisition, the program executes a routine to find the main peaks, followed by a routine that performs smoothing with an algorithm based on penalized least squares strategy and getting as a result smoothed data for further processes. Such a strategy provides the possibility of using *n*th-order decomposition on the experimental data. Then, a routine based on the Rachinger correction subtracts the $k\alpha_2$ radiation. To obtain the microstructural information, a linear interpolation method can obtain FWHM values for each peak; or by a routine based on a trapezoidal method, which can be executed to calculate the integral breadth values. The program is able to perform either Cauchy/Cauchy or Cauchy/Gaussian line profiles in units ($\delta 2\theta$ units). If the profile is assumed to be Cauchy, the following relationship holds $\beta^* = 1/\tau + 2\zeta d^*$, and where $d^*=(2\sin\theta)/\lambda$ is the inverse of the scattering vector, ζ and τ are the apparent strain and sizes, respectively. Finally, the broadening profiles can also be approximated by Gaussian functions. The routines are written in both GNU Octave (version 4.2.1) and Julia (version 0.6.2) programming languages for easy and fast implementation. Gold thin films were prepared from PVD methods. Diffractograms of samples were acquired on a Bruker X-ray D2 Phaser powder diffractometer using β-filtered, Cu-Kα radiation (λ= 0.154 nm; 30kV, 10mA) (Fig. 1A). LPA analyses are showed in Figures 1B and 1C. Diffract.EVA suit package program (version 4.1.1; Bruker AXS Inc., Madison, WI) was used for data comparison regarding FWHM and Integral Breadth values (Fig. 1D). A complete analysis of microstructural parameters (e.g., average crystallite size and the percentage of lattice strain) obtained with our routines is discussed along the presentation.

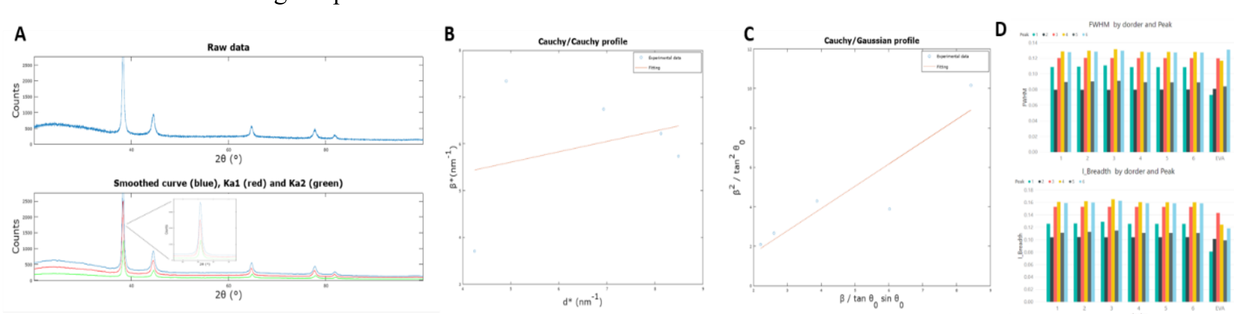

Figure 1: (A) Raw gold thin film XRD data vs smoothed data and smoothed data compared to $k\alpha_1$ $k\alpha_2$ radiation contributions. (B) Anisotropic analysis of Integral Breadth considering a Cauchy/Cauchy line profile. (C) Anisotropic analysis of Integral Breadth considering a Cauchy/Gaussian line profile. (D) Performance comparison when varying decomposition *n*th-order in smoothing routines.

References: (1) F.F. Contreras-Torres, et al., J. Nanosci. Nanotech, 17 (2017) 939; (2) D. Cavazos-Cavazos and F.F. Contreras-Torres J. Phys. Chem. Solids 110 (2017) 36.