# Combined X-ray Texture-Structure-Microstructure Analysis Applied to Thin Ultrastructures: A Case Study on $Pb_{0.76}Ca_{0.24}TiO_3$

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The complete determination of the microstructure of a thin film includes analyses of the crystallographic preferred orientations and structures, crystallite sizes, microstrains, crystalline ratio, layer thicknesses ...

Most of the microstructural details can give specific contributions on the X-ray diffraction signals, but their combination in a single diagram, as in the case for most of the elaborated materials, makes their analyses particularly difficult. Unfortunately, one cannot avoid treating all of these contributions as a whole, since they are inter-dependant. For instance, refining the texture without knowing the exact structure can give rise to artefacts in the texture treatment, and inversely. The presence of several phases can furthermore make individual methods to cancel because of strong peak overlapping.

Only recently, a methodology has been developed which combines the principal aspects of the microstructural contributions to neutron diffraction data. When using X-rays, such an analysis is even more complex, mainly because of the high absorption coefficients of X-rays. Most of these problems are now resolved, and results of the so-called "combined" approach are presented here, on a Ca-modified $PbTiO_3/Pt/TiO_2/SiO_2/Si$-(100) ultrastructure elaborated by spin-coating.

The methodology uses measurements on a 4-circle x-ray diffractometer equipped with a curved position sensitive detector. 2θ diagrams are acquired in aproximately 700 different tilt and azimuth orientations of the sample. A first cycle using a Rietveld algorithm is operated cyclically on all diagrams with starting parameters and taking into account experimental resolutions (including defocusing), including a layer formalism. Then, a Le Bail extraction is used to provide integrated intensities as input for a quantitative texture analysis by a WIMV-like algorithm. Resulting parameters are used for a second Rietveld cycle and so on.

This methodology allows the structure parameters to be determined (here the cell parameters), with particle sizes, microstrains and layers thickness, with the quantitative texture, for both the PTC and Pt layers. It allows for instance the separation of the <100> and <001> components of the texture, of considerable importance for macroscopic physical properties.