X-ray textural and microstructural characterisations by using the combined analysis approach for the optical optimisation of micro- and nano-structured thin films

M. Morales[1*], D. Chateigner[2], L. Lutterotti[3]

[1] *SIFCOM-ENSICAEN, CNRS, Université de Caen Basse-Normandie, 6 bd du Marechal Juin, 14050 Caen Cedex 04, France*

[2] *CRISMAT-ENSICAEN, CNRS, Université de Caen Basse-Normandie, 6 bd du Marechal Juin, 14050 Caen Cedex 04, France*

[3] *Dipartamento Ingegneria dei Materiali, Universita di Trento, via mesano, 38 Trento, Italia*

**magali.morales@ensicaen.fr*

Thin film X-ray analysis is nowadays more and more confronted with major problems when the elaborated films exhibit desired or not preferred orientations. Indeed the intensity variations of the X-ray reflections are dependent simultaneously on the texture and the structure, and the texture is *a priori* unknown. Moreover, these difficulties are emphasized when (i) the films are nano-structured with a lot of inter and intra-phases peaks overlaps, (ii) when the crystallites have anisotropic shapes (different inter and intra-phases overlaps with hk□), (iii) when the samples are composed of several layers of different phases (with *a priori* different textures) and finally (iv) when residual stresses are evidenced (different overlaps and position peaks with the sample orientation). Moreover, volume and absorption corrections become necessary (as they bias structural and textural approaches) and which are dependent on the film true absorption coefficient, *a priori* unknown due to the strong porosities exhibited in general in the elaborated films.

A new methodology called "combined analysis" has been recently developed in order to implement the determination of all these parameters accessible by x-ray diffraction [1] and necessary in order to optimize the physical properties of the films. This technique is based on cyclic refinements of the diffraction diagrams measured for different sample orientations and by using the Rietveld Method. The texture is then modelled through the refinement of the orientation distribution function (ODF) by using the E-WIMV method, the crystallite sizes and shapes by using the anisotropic Popa formalism and the residual stresses through the film elastic constants refinement from the ODF by the geometric mean of the elastic tensor. The film thicknesses are also refined by using the absorption corrections.

This methodology requires however a lot of data in order to take into account all sample orientations necessary for a good ODF determination, i.e. around 1000 2θ diffraction patterns. For their rapid acquisition it appears then necessary the use at a minimum of linear detectors.

The use of this combined approach in order to optimize the optical properties of micro- and nano-crystallised (nc) thin films will be illustrated through three examples:

- nc-Si crystallites embedded in an amorphous SiO_2 matrix and deposited on glass or single crystals [100]-Si substrates, as an example of the texture and the film thicknesses determination coupled with the presence of anisotropic Si crystallite sizes [2];
- nc-SiC crystallites embedded in an amorphous SiC matrix and deposited on glass or single crystals [100]-Si substrates, as an example of the texture determination with anisotropic SiC nanosizes and the presence of two polytypes of SiC [3];
- Cr^{2+}:ZnSe thin films deposited on glass substrate to show the actual technique limitations when stacking faults and strains are evidenced.

[1] J. Ricote, D. Chateigner, M. Morales, M. L. Calzada and C. Wiemer. Thin Solid Films **450** (2004) 128.

[2] M. Morales, Leconte, R. Rizk and D. Chateigner, Journal of Applied Physics **97** (2004) 034307.
[3] H. Colder, R. Rizk, M. Morales, P. Marie, J. Vicens, Journal of Applied Physics **98** (2005) 024313