CHARACTERIZATION OF SUB-MICRON THIN FILMS AND MULTILAYERS BY ENERGY DISPERSIVE X-RAY FLUORESCENCE

I.Queralt[1], E.Marguí[2], X.Llovet[3], J.Pujol[4] and J.F.Piniella[5]

[1] Laboratory of X-ray Analytical Applications (LARX). Institute of Earth Sciences "Jaume Almera", CSIC. Solé Sabarís s/n.ES- 08028 Barcelona, Spain.
[2] Department of Chemistry, University of Girona, Campus Montilivi, ES-17071 Girona, Spain.
[3] Universitat de Barcelona, Serveis Científico-Tècnics, Lluís Solé i Sabarís, 1-3, ES-08028 Barcelona, Spain.
[4] Fischer Instruments SA, Almogàvers St., 157, ES-08018, Barcelona, Spain.
[5] Autonomous University of Barcelona, Crystallography Unit. ES-08193 Cerdanyola del Vallès, Barcelona, Spain.

The growing importance of thin-film technologies requires precise, accurate and reliable methods for the analysis and characterization of layer properties, as the quality and adequate technical use of many modern materials strongly depend on the thickness and homogeneity of the deposited layers. Different analytical techniques (EPMA, TEY, XPS, GIXRD, XRR, RBS, EDXRF, WDXRF …) have been checked for determining the thickness of coatings at levels of some microns or even for the evaluation of ultrathin layers.

X-ray fluorescence has been routinely applied for the quantitative elemental analysis of different inorganic materials, and it has been also accepted as a common analytical technique for layer thickness measurement in many classical industrial applications, especially for determination of metal coatings. Several international norms [1,2] are based on X-ray fluorescence (XRF) spectrometry, providing the basis for the assessment of the quality control in coating industry. Different XRF configurations, including benchtop and portable XRF instrumentation have been applied to provide information on the geometry and composition of layered and multilayered materials. X-ray fluorescence does not provide direct measures of coating layer thickness. The basic instrument response that is measured is the total X-ray intensity or the counting rate (counts per second, cps) of the spectral lines from the characteristic elements in the layer (emission method) or the intensity of lines from elements existing in the substrate (absorption method). The use of XRF instrumentation is supported by a greater penetration depth and the possibility to reduce or enlarge the focal spot from few tens to some thousands of micrometers thus obtaining a representative average of layer properties both at the surface and in-depth layers.

In the present work we undertake the study of different kinds of thin layers and multilayers by μ-EDXRF under the criteria of validation procedures, keeping in mind the usual protocols for any analytical procedure. Therefore, in accordance with the usual guidance for validation of analytical procedures we considered several validation characteristics. For instance, specificity, linearity, range, accuracy and precision must be considered. Additionally we also checked the homogeneity of layered samples by elemental mapping. Among others, we used for the study Au-plated multilayered metallic samples, yttria-stabilized zirconia (YSZ) films deposited on pure silicon, gallium nitride films on sapphire substrate [3] and zirconia nanolayers on steel.

[1] ASTM, Standard B568-90, Standard Test Method for Measurement of Coating Thickness by X-ray Spectrometry, American Society for Testing and Materials, Philadelphia, PA, 1990.
[2] International Standard ISO 3497 Norm, Metallic Coatings – Measurement of Coating Thickness – X-ray Spectrometric Methods, Third edition 2000-12-15, Geneva, Switzerland, (2000).
[3] Queralt I, Ibañez J., Marguí E. and Pujol J., Thickness measurement of semiconductor thin films by energy dispersive X-ray fluorescence benchtop instrumentation: Application to GaN epilayers grown by molecular beam epitaxy. Spectrochimica Acta Part B, 65:583:586, 2010