

Combining Scanning Probe Microscopy and X-Ray Spectroscopy to Obtain Simultaneously Surface Topography and Chemical Mapping

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Non destructive tools providing elemental and chemical analysis at high resolution are necessary for life and physical sciences. For example electronics or glass industry needs in-lab tools for material processing and control (RRAM, FeRAM, smart materials, solar cells) [1].

Near Field Microscopes are powerful tools for surface topography analysis at nanometric lateral resolution. But, these equipments cannot provide chemical mapping of the analysed surface.

X-Ray Spectroscopies are fine analysis techniques providing chemical and structural properties of a material, based on the spectroscopy of the emitted photons or photoelectrons. They require a high brightness excitation X-Ray source, synchrotron beam, to irradiate the sample. The lateral resolution is about 1 μm , but it is not possible to image simultaneously the sample surface neither to position the X-Ray beam on a peculiar micro or nano object. Among these techniques, XEOL spectroscopy (X-Ray Excited Optical Luminescence) deals with the analysis of visible photons emitted from the surface of the sample.

We have designed and fabricated a new characterization tool combining X-Ray Spectroscopy and Shear Force Microscopy and working at ambient conditions, allowing surface topography measurement simultaneously to chemical mapping [2,3]. The probe of the microscope is a sharp and low aperture optical fiber used to detect the XEOL signal of the sample under synchrotron irradiation. Simultaneously, it is possible to obtain the topography of the sample surface. Thus, the instrument is able to image the surface and to localize a peculiar object that can be further chemically analyzed by XEOL analysis.

In this work, we present the results obtained on the ID03 line at ESRF. First experiments were performed on a ZnO layer deposited on a silicon sample. Figure 1a) shows the conventional XEOL spectra acquired on ZnO powder and on the ZnO layer. Figure 1b) shows the XEOL spectrum of the same layer recorded in near field using our apparatus. The spectra acquired either in far or in near field are in good agreement with the ZnO powder reference spectrum. Moreover, collecting the XEOL signal in near field increases significantly the lateral resolution of the XEOL technique, which is now limited only by the aperture of the optical fiber.

Further characterizations were performed on ZnWO₃-ZnO layer co-sputtered on a silicon sample. Figure 2 shows both topography and luminescence cartography of this layer recorded simultaneously. In the upper figures (a, b, c, d) the sample topography is presented. We show in the figures (e, f, g, h) the corresponding luminescence cartography obtained respectively, from the left to the right, before and after the Zn-K edge, as well as before and after the W-K edge. The acquisition time was 45 minutes per twin image topography-light. From (f) and (h) images, chemical mapping of the sample, i.e. regions rich in Zn or W can be defined.

Replacing the optical fiber of our microscope by an X-ray monocabillary, it is possible to collect the X-Ray Fluorescence (XRF) in near field instead of the XEOL signal. An Energy Dispersive X-Ray analyser is thus used to carry out the spectrometry of the fluorescence signal. Up to now, we have demonstrated the feasibility of this concept and that a resolution of 1 μm can be reached using a laboratory X-ray excitation source (rotating anode), while a resolution of less than 100 nm is expected using a brighter source (synchrotron environment). Of course the sample topography can be obtained simultaneously to chemical analysis.

References

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[2] D. Pailharey et al. Journal of Physics: Conference Series 93, Functional Materials and Nanotechnologies, 2007

[3] S. Larcheri et al. Review of Scientific Instruments **79**, 2008

Figures

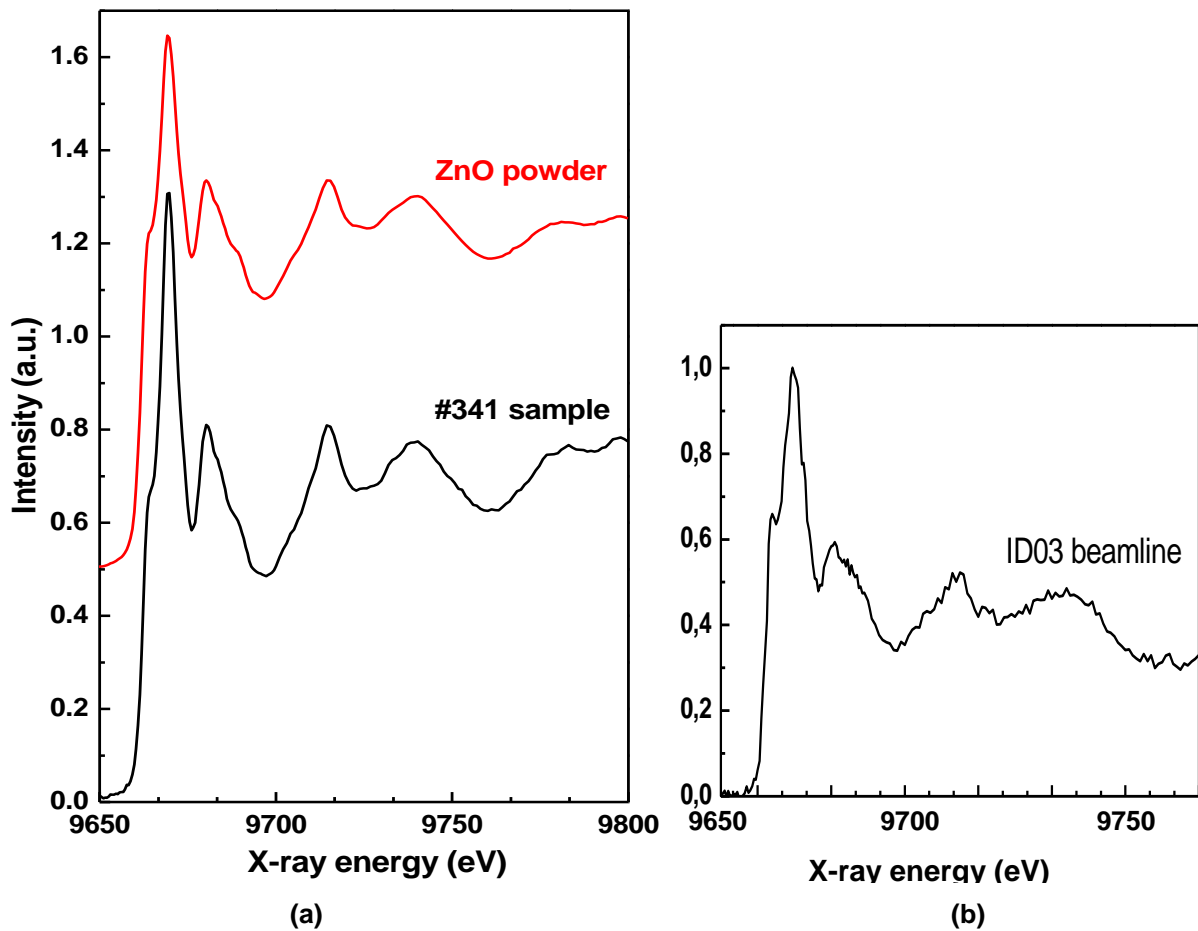


Figure 1 : (a) XEOL spectra of ZnO powder (top) and of a ZnO layer sputtered on silicon sample (bottom). (b) XEOL spectrum of the same ZnO layer recorded in near field with the sharp and low aperture optical fiber, probe of our shear force microscope.

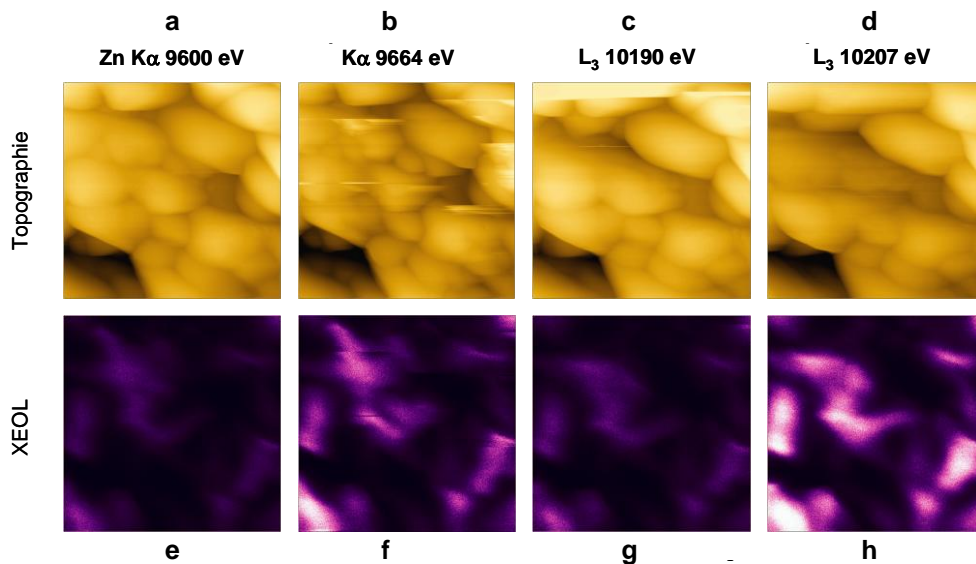


Figure 2 : (a-d) topography of a sample coated with the ZnWO₃ + ZnO layer (2 x 2 μm²). (bottom) corresponding visible light image under illumination by X-ray beam from left to right below (e) and above (f) the Zn threshold (9.6 keV) and below (g) and above (h) W threshold (10.2 keV).