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Near field X-ray spectromicroscopies: new tools for nanoscience

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In the last years, the X-ray absorption (XAS) techniques have undergo remarkable development: (i) experiments with unprecedented femtometer accuracy, under extreme conditions of high pressure and temperature [1], (ii) experiments with nanoscale lateral resolution [2]. Nevertheless, investigations of complex nanostructured materials used in modern technologies require special X-ray experimental techniques able to imaging simultaneously topography and chemical mapping (X-ray analysis of matter) on the nanometer scale.

Near Field (NF) X-ray Spectromicroscopy (FF illumination and NF detection) is a fully new approach for the detailed investigation of nanostructures down to the nanometer level. The extremely high lateral resolution of Local Probe Microscopies (LPM, AFM,STM) makes them among the most largely used in nanoscience. However, these tools suffer of a lack in chemical sensitivity. On the other hand, *far field X-ray spectroscopy* probes the chemical and structural properties of materials. A combination of X-ray spectroscopies and LPM is the ideal answer to many problems in nanosciences. This report highlights the most important contributions which were held in the combination of X-ray spectroscopies and LPM techniques.

The basics of such approach are circulating since years. The first observations of core-level photoelectrons generated by X-ray irradiation of the tip-surface region of STM have been published by Tsuji [4]. Ishii [5] has measured the capacitance XAS signal with a metal tiny electrode. The combination of XAS and scanning near-field optical microscopy (SNOM) as a local detector was proposed by Purans [6], while a combination of XRF technique and LPM with a cantilever, having a hole of 100 nm, as a *collimator* of X-ray beam was proposed by Nagamura [7].

First STM and SNOM experiments under *focused* synchrotron-radiation (SR) were performed at ESRF on the microbeam line ID-3 [8]. Detailed STM study using soft SR X-rays was performed by Matsushima et al. [9]. A STM dedicated to in situ experiments under the irradiation of highly brilliant hard-X-rays of synchrotron radiation has been developed by Saito et al. [10] and a current modification was detected at the absorption edge with a spatial resolution of the order of

10 nm. Finally, Ishii and Hamilton et al. [11] has combined electrostatic force microscopy (EFM) with tunable synchrotron x-ray source excitation.

Further progress we have achieved in the framework of the European X-TIP project by the focusing SR beam to increase the density of the incident photons. X-ray optics at third generation Synchrotron Radiation facilities have lead to the stable production of X-ray microbeams with extremely high photon densities making this approach feasible. We have started with three types of experiments: (i) XAS-AFM: X-ray excited secondary electrons detection by conductive tip in AFM mode; (ii) XAS-SNOM: X-ray excited optical luminescence (XEOL) detection by SNOM in AFM mode; (iii) XAS-SCM/AFM: X-ray excited capacitance or/and photoconductivity of sample detection by conductive tip in SCM, KFM or AFM mode.

The new instrumentation developed within this project offers the possibility to carry out a selective structural analysis of the sample surface with the subwavelength spatial resolution determined by the SNOM probe aperture. In addition, the apex of the optical fibre plays the role of a topographic probe, and chemical and topographic mappings can be simultaneously recorded.

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