Preliminary Report for IN-564 experiment, 14-18th february, 2007

Measurement of the lateral resolution of the NanoESCA instrument in XPEEM mode and applications in two selected case studies

This experiment was the continuation of the first round of IN-564 which took place from august 22nd to august 28th, 2006 and which was dedicated to the commissioning of the NanoESCA XPEEM spectromicroscope purchased by CEA using synchrotron radiation. Indeed in order to reach lateral resolutions below 150nm in energy-filtered core-level imaging, XPEEM needs bright soft x-ray sources.

During the first round, a lateral resolution of 120 nm with core-level was demonstrated [1]. This is already an excellent result if one considers that the ID08 beamline is not optimized for fine focusing which lowers considerably the effective photon flux (ie, within the field of view), typically in the order of 3.6×10^{11} ph/s/0.1% BW. The result had been obtained on a sample prepared in our laboratory with FIB-etched patterns, which presented the drawback of not producing perfectly sharp chemical transitions [1].

During this round we used a sample especially dedicated to the calibration of the lateral resolution of imaging instruments and currently under certification [2]. It is a cross-section of an epitaxially-grown multilayered structure consisting of alternate GaAs/AlGaAs layers organized to make AlGaAs/GaAs/AlGaAs gratings of different thicknesses, from 700 down to 25 nm. Using these gratings, a true resolution of the microscope could be measured, as well as edge resolution using the GaAs/AlGaAs bilayer structures, without suffering from imperfectness of the edge quality.

After suitable *in-situ* surface preparation, adjustments of the NanoESCA optics were performed under more stringent conditions regarding lateral resolution than the ones used during the first round, the whole process taking about 3 shifts. We recorded energy-filtered images at the photoemission threshold at high energy resolution and 500eV photon energy; Fig. 1 displays one of these image, these images taken at with photoelectron energy where the optimal work-function contrast takes place. The image was recorded with secondary photoelectrons of 6 eV kinetic energy above the Fermi level of the sample, which is above the work-function of AlGaAs but in the low intensity tail of the secondary electron distribution for GaAs. Therefore at this electron energy, AlGaAs layers appear bright whereas GaAs layers looks dark.

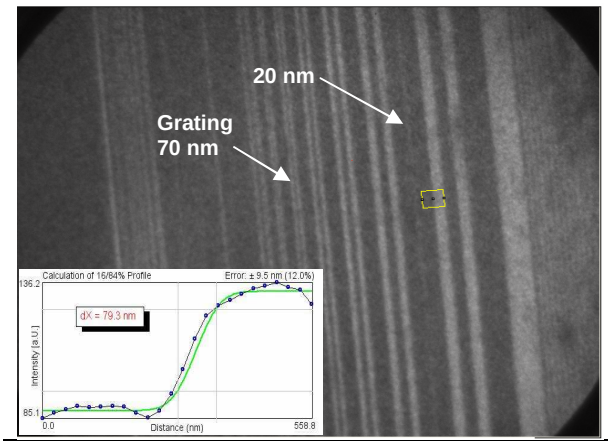

Fig. 1- XPEEM image taken above the photoemission threshold of AlGaAs (E-Ef= 6 eV). Dark layers are GaAs; bright layers are AlGaAs.

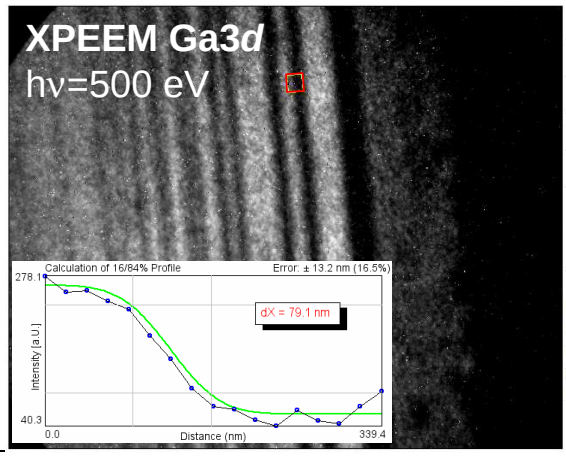

Fig. 2- XPEEM Ga3*d* (E-Ef=379 eV) core-level image with NanoESCA.

This example illustrate nicely the capabilities in energy filtering of the *NanoESCA* spectromicroscope. On fig. 1, it can be clearly seen that the thinnest layer of 20 nm (marked with the arrow) can de detected (point resolution). It seems that the **70 nm** grating can be resolved, which is confirmed by the intensity profile (inset) taken for estimating the edge resolution.
For core-level imaging, we used the Ga 3*d* core-level for its high cross-sections at 400 eV. We recorded 25 images for a better statistic and superimposed them as shown in fig. 2. Preliminary results concerning the lateral resolution of this image seem to confirm that the edge resolution obtained in this core-level image is **below 100 nm**, as shown by the intensity profile displayed in the inset.

These good results in the lateral resolution were confirmed with two case studies performed during the beamtime. The preliminary results are shortly described below.
The first one concerns XPEEM images on *single* silicon nanowires dispersed on gold patterns. The diameter of the nanowires is 150 nm. Fig. 3 displays the O1*s* XPEEM core-level image. The acquisition time is 15 min. The intensity from O1s photoelectrons is clearly localized on the nanowires, evidencing surface oxidation. The inset displays an image taken at the photoemission threshold (E-Ef=4.6 eV), showing that the observed nanostructure is not a single, kinked wire as one could derived from the core-level image but two isolated wires. In fig. 4 we display the localized threshold photoemission spectra *along the NW*. They show the presence of a *double photoemission threshold*, one intrinsic to the Si NW with the characteristic work-function of 3.6 eV expected [3] another close to that of the gold substrate . This is indicative of gold partially covering the surface of the NW as a result of the VLS process, in agreement with recent results [4].
The second case studies concerns polycrystalline copper with palladium at the surface. Pd is heterogeneously distributed depending on the local crystalline orientation of the copper grains. We obtained images at the photoemission threshold as displayed in Fig. 5. Spectromicroscopy at the threshold enables to investigate local variations of the work-function due to this heterogeneous distribution of Pd. XPEEM core-level images with Cu3p and Pd3d photoelectrons were also acquired successfully. What we could confirm is the heterogeneous distribution of Pd. These data are currently processed more accurately.

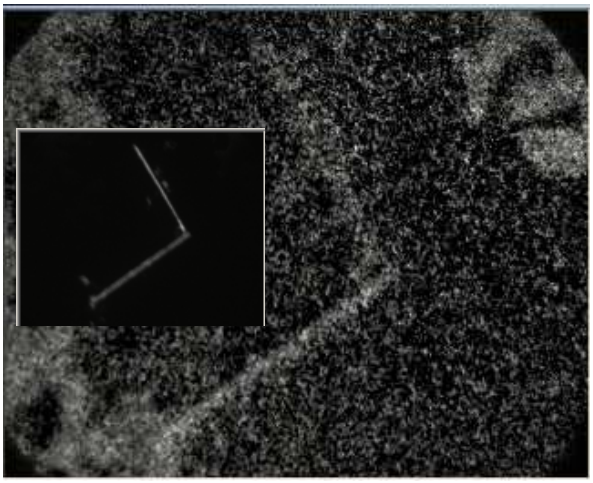

Fig. 3- XPEEM O1s core-level image on single silicon nanowires (150 nm diameter); inset : image at the photoemission threshold (field of view 18 µm).

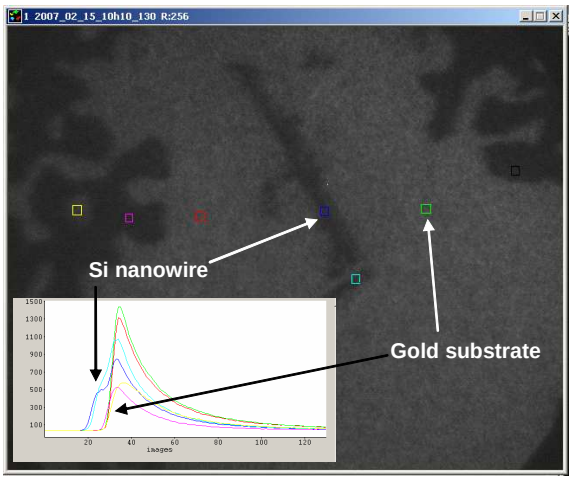

Fig. 4- XPEEM image at the photoemission threshold and generated nano-spectra (inset) over the marked areas.

Fig. 5- XPEEM image at the photoemission threshold of polycrystalline copper surface with Pd at the surface (field of view 18 μm).

[1] O. Renault, N. Barrett et al., Proc. 5th LEEM-PEEM international conference, 2006, to be published in *Surf. Sci.* 2007.

[2] M. Senoner *et al.*, *Surf. Interface Anal.* 36, 1423 (2004).

[3] B. Zeng, G. Xiong, S. Chen, S. H. Jo, W. Z. Wang and Z. F. Ren, *Appl. Phys. Lett.* **88**, 213108 (2006).

[4] J. B. Hannon, S. Kodambaka, F. M. Ross and R. M. Tromp, *Nature* **440**, 69 (2006).