X-Tip Joining X-Ray spectroscopies to Local Probe Analysis: a new tool for nanoscience

Dhez Olivier[1], Rodrigues Mario[2-3], Comin Fabio[2], Felici Roberto[1], Chevrier Joel[3]

[1]OGG-CNR-INFM, Grenoble, France, [2]ESRF, Grenoble, France, [3]UJF/CNRS, Grenoble, France

Abstract. With the fast development of the nano-scale science, the need of tools to be able to image the sample and bring the region of interest to the X-ray beam is essential. We demonstrate the possibility to utilize high resolution imaging capability of the Scanning Probe Microscopy (SPM) to image and align the sample with the X-ray beam, and the possibility to record the photoelectrons emitted by the sample.

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Our goal is to combine the high lateral resolution imaging capability of Scanning Probe Microscopy (SPM) with the high penetration power and high elements sensitivity of X-rays. We will show the ability of our home build microscope to perform AFM imaging on a beamline sample holder, align the AFM tip with the X-ray beam, and perform total electron yield detection (TEY) with the AFM tip

We build a microscope that in term of noise and vibration is compatible with the rough environment of the X-ray beamline. Our microscope is based on piezoelectric quartz tuning forks oscillator, using the shear force detection mode [1]. The high-quality factor of the tuning fork allows to perform non-contact experiment in shear mode, even in air. We use chemical etched tungsten tip glued on one sprung of the tuning fork. The tip apex vary from 10 to 50 nm. In our setup we decide to have the tip fixed in position and to archieve all the displacement by the sample. A coarse displacement of 4 mm with a resolution of 25 nm is obtained by using a set of Attocube motor. The scanning ability and fine displacement of 9 µm travel with 0.02 nm resolution and tip-sample regulation are performed using a microtritor from PiezoJena. The microscope electronic control and data acquisition is performed by a RHK Technology SPM 1000 Scanning Probe Microscope Control System.

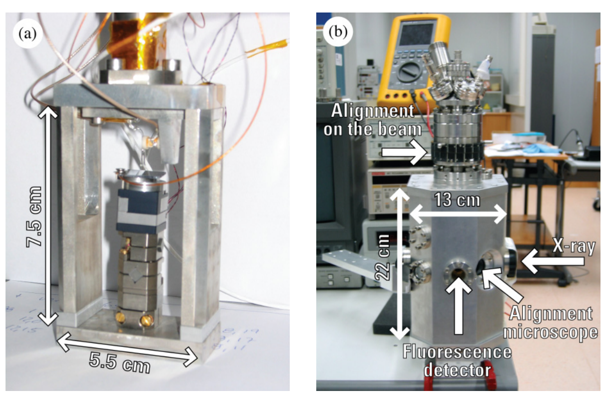

FIGURE 1. (a) View of the microscope composed of 3 Attocube coarse motors and a PiezoJena microritor scanner. (b) General view of the vacuum chamber that old the microscope.

The whole microscope is enclosed in a vacuum chamber (Figure 1) that can reach the 10^{-8} mbar range. The chamber ports gives us the ability to measure the X-ray fluorescence signal, the transmitted X-ray and also to place a

VLM (visual light microscope) to pre-align the microscope on the beam. The whole microscope can be manually pre-align on the beam by the use of a port-aligner.

The Atomic Force Microscope.

Our microscope is able to perform imaging on an X-ray beamline. The figure 2 show an AFM image, obtained in shear mode of Ge dots on Si. We can achieve a resolution in (X,Y) of 25 to 50 nm depending on the level of noise and vibration surroundings. Even some tests have been made to check the ability of keeping vertical resolution (tip-sample distance constant) of a few tens of nanometers and move the whole microscope on a standard Huber goniometer.

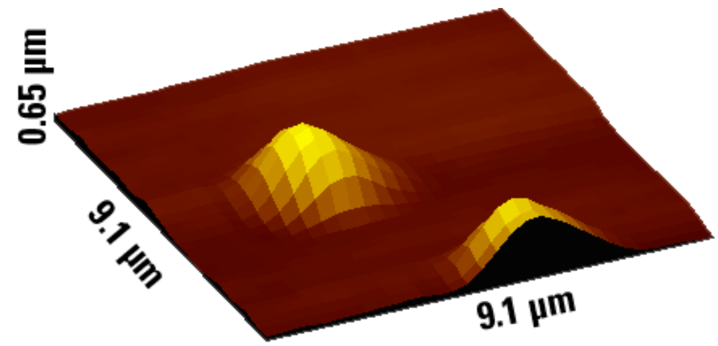

FIGURE 2. AFM image of Ge dots on Si obtain on ID01 Beamline at the ESRF.

We can image and align the AFM metallic tip and the X-ray beam. Different possibilities are offered: measuring the current produce by the interaction of X-ray with the metallic tip, measuring the transmitted intensity or measuring the fluorescence signal of the tip. To measure the tip current we need to use a lock-in amplifier due to the very low current. The figure 3a show the current and the phase, "phase" being for us the difference between the measured signal and the reference signal. On the figure 3b we show evolution of the fluorescence signal of the sample (Ge dots in Si), and the fluorescence signal of the tip. In this case, the tip was set above the sample by about 500 µm. When we move the whole chamber vertically we first observe the fluorescence signal of the tip (plain line) then the fluorescence signal of the Ge (dash line).

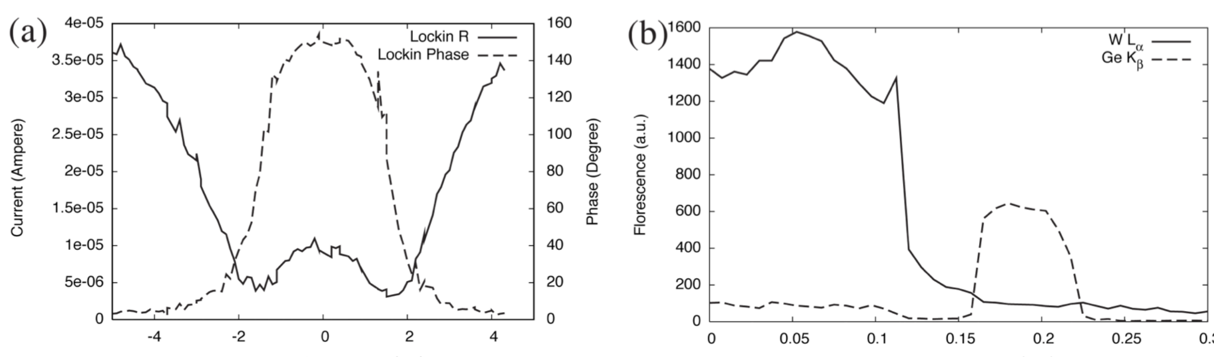

FIGURE 3. (a) Current and phase recorded by the lock-in amplifier though the AFM tip. (b) Fluorescence signal from the W tip and the Ge dos of the sample.

The figure 4a show a map of the current measured by the tip, and the figure 4b is a map of the transmitted beam. These two figures demonstrate the highest sensitivity when using the sharp W tip to detect the beam, than using the absorption image.

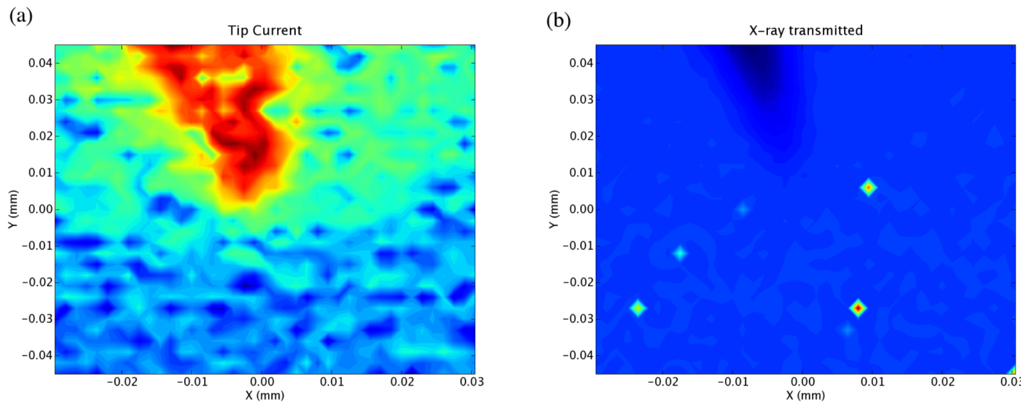

FIGURE 4. (a) Current measure by the metallic tip. (b) Transmitted intensity through the microscope.

After demonstrating our ability to obtain good AFM image on a beamline sample table, to align the microscope on the X-ray beam and selecte the region of interest for the X-ray studies, we are discussing below the possibility to record total electron yield signal with this microscope.

Total Electron Yield detection by an AFM tip

They are several advantages of using a metallic tip as a detector for the TEY (total electron yield). First we are not sensible to the beam position vibrations, because the size of the tip, typically the apex is in the order 50 nm, is smaller than the focus beam size, which is typically microns. Secondly, the resolution is not independent of the beam size, the area of electrons collection will depend on the tip size.

Figure 5 show the general setup used to perform our TEY experiment and the X-ray beam chopper used for the lock-in amplifier.

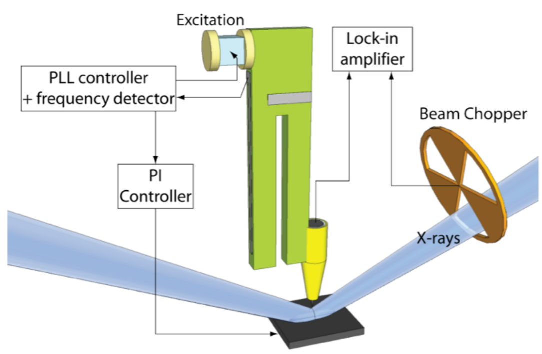

FIGURE 5. Geometry of the experiment showing the tuning fork and the beam geometry with the locking system.

In order to estimate the expected detectable current that we will have to detect, we performed some Monte Carlo simulation using the Penelope code[1]. The figure 6a show the calculation of the TEY current for a Ge dot of 36 nm of radius on a 10 µm thick Si wafer with an X-ray flux of 10^{12} ph/s, assuming that all the X-ray photons are concentrated on the Ge dot. In these conditions we obtained a jump of 28 pA when crossing the Ge K absorption edge.

[1] Penelope code (http://www.nea.fr)

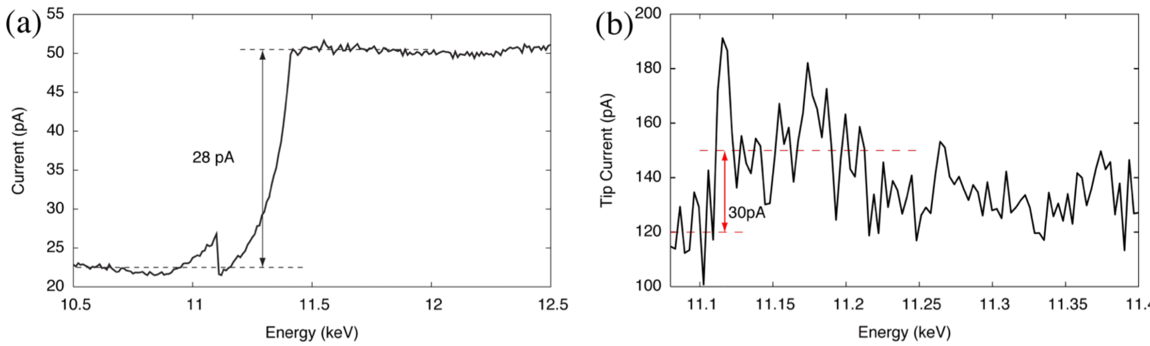

FIGURE 6. (a) Result of the Penelope simulation for the photoelectric current calculated for a Ge dots on Si when crossing the Ge K absorption edge. (b) Current record by the tip when crossing the Ge K absorption edge

This is to compare to the result shown on the figure 6b corresponding to the experiment performed at the ESRF beamline ID22. For this experiment we used a focused beam of 2 x 3 μm^2 with an incident flux of 6.10^{11} ph/s. The sample-beam grazing incident angle is in the order of 5º. The figure 6b shows the TEY current measured by the tip when crossing the Ge K absorption edge. The curve indicates that the present measure is quite noisy even with the use of a lock-in detection. During this experiment the small displacement stage was not implemented, our lateral positioning was very bead. So we don't really know the position respectively to the Ge dot. This may explain the high level of noise. We can nevertheless distinguish a jump at the energy of the Ge K absorption edge. The jump value of this edge is in the same order than the one calculated by the Monte Carlo simulation. We have to push forward our test, but it seems that the use of the TEY detection by an AFM tip is very challenging.

Conclusion

We have shown that is now possible to perform AFM topological images on a standard X-ray beamline environment. That is bringing the possibility to use an AFM to select the region of interest with a nanometric precision and in addition to align it with the X-ray beam.

For the TEY detection the current level is very low. Our Monte-Carlo simulation show that the current level is in the order of Pico Ampere The signal noise ratio may be improve by the use of coaxial isolated tip, that are now under development,.

The future perspectives are the use of this kind of microscope to perform nano-manipulation and nano-interaction with matter.

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