Optimal focusing system of the Fresnel zone plates at the Synchrotron SOLEIL NanoARPES beamline

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Abstract. ANTARES beamline (BL), operating at very low photon energies, is a new soft X-ray scanning microscope recently built at SOLEIL Synchrotron, that offers a spectroscopic non-destructive nano-probe to study advanced materials. It combines a set of Fresnel Zone Plates (FZP) able to focalize the beam spot up to a few tenths of nanometres with a stable and precise sample nanopositioning (<1 nm). High energy-, angular- and spatial- resolution allow accurate electronic and chemical imaging combining angle-resolved photoelectron spectroscopy (NanoARPES or k-nanoscope) and core level detection by using both photoemission and X-ray absorption. Here, we report our latest results related to the optimization of the post-focusing mirrors system as well as its impact on the ultimate spatial resolution of the whole microscope.

1. Introduction

Recently, a fast progress in modern microscopic methods has been realised to image nano-objects and heterogenous samples in nano and mesoscopic scale. In particular, the X-ray spectromicroscopy, which combines the abilities to get the spectroscopic information by X-rays excitation of the samples with the microscopy. However, beyond the achievement of Angstrom spatial resolution, the challenge still remains in order to consolidate lateral manometric resolution with high resolution energy-resolved spectroscopic methods able to produce chemical and electronic imaging. Part of these challenging objectives have been already accomplished by the study of core levels and their chemical shifts using scanning photoelectron microscopy (SPEM) and X-ray scanning transmission microscopy (SXTM) techniques. However, these two powerful methods are no skilful in producing electronic imaging of the valence band states close to the Fermi level. Even if these electrons are essential determining the transport, magnetic and chemical properties of the investigated samples.

Even if we are strongly concerned by core level imaging, our approach has been in addition to adapt the competences of ARPES to the FZP and scanning system set-up to produce electronic imaging. The result is a new sophisticate and versatile nanoscopy named nanoARPES our k-nanoscope[1, 2, 3]. This technique is one of the most direct methods of studying the electronic structure of solids and is the only truly momentum-resolved probe, which is essential for the investigation of low dimensional and strongly anisotropic systems. By measuring momentum and kinetic energy of the electrons photoemitted from a sample illuminated with radiation of energy larger than the material work function, it is possible to gain information on both energy
and the momentum of the electronic excitations close to the Fermi level, inside the solid. In this contribution we present the latest electronic and chemical imaging of nano-structured samples, as well as the repercussions of the BL focusing post-focalization on the accuracy and precision of the NanoARPES images.

2. Layout of the focusing system and results

The ANTARES BL was remarkably designed for NanoARPES experiments, core levels imaging and x-ray absorption spectromicroscopy in the photon energy range between 10 and 1000 eV using lineal and circular polarized light. The BL includes two insertions devices and the main components are: its pre-focalization optics, a plane grating monochromator (PGM) and two post-focalisation optics each of one is a Pseudo-Wolter, for more details see [1, 2, 3] and references therein. Each independent pseudo-Wolter system is composed by a set of spherical and toroidal mirrors in horizontal derivation. One pseudo-Wolter is able to focus directly on the sample while the second one its focus is coincident with position of the pinhole at the Zone Plate object plane, see Figure 1.

![Figure 1. Schematic representations of the Pseudo-Wolter post-focusing and FZP systems. (a) The Figure presents the focalisation system of the ANTARES BL with two pseudo Wolter systems mounted in an elevator system that allow easily to select the illumination focus at the pinhole (Wolter I) or at the sample (Wolter II). Panel (b) shows schematically the focalization of the X-ray of the BL using the FZP The FZP is fixed on a three-axises manipulator with high precision and stability, which can set the FZP in or out the beam path. Besides, the optical focalising system also consists of a pinhole and an Order Selection Aperture (OSA) system placed between the FZP and the sample, which suppresses unwanted diffraction orders. The pinhole is situated at the (object) or (source) position and the sample at the (image) position, of the optical system, espectively (see Fig. 1b). The distances between the FZP and the object and the image are called p and f, respectively. For ANTARES BL the distance p is close to ≈1.5 m, although it depends to a slight extent on the photon energy. The FZP, as a diffracted lens, focusses the X-rays at the image plane, placed at a distance f from de FZP. The primary aim of the BL focalization system is to define an almost perfect infinitely circular point source (or object) because if the shape of the source is distorted, the generated image will be also distorted with the same shape, as it has been schematically indicated in Fig. 1b. Most importantly, the size as well as the shape of the image in plane B is defined by the demagnification, which is a function of the magnitudes p and f . Effectively, the simplified expression of the lowest spatial resolution for a typical FZP is given by [4].

$$\text{Resolution} = \frac{1}{2\pi} \sqrt{\frac{\lambda}{\sin\theta}}$$
\[ \delta = \frac{1.22 \Delta r}{m} \]  

Figure 2. Optimization of the Pseudo-WOLTER Panel (a) shows the shape of the light point at the pinhole, plane A in Fig. 1b, when the positioning parameters of the pseudo-Wolter are not optimized (i.e. Roll, Pitch and Yaw positioning). Panel (c) image of rectangular nanowires of 1um long and 200 nm width measured with the point source illustrated in panel (a). Panel (c) shows the shape of the beam as in panel (a) once the Pitch and Yaw parameters have been optimized. In panel (d), shows the image of ordered gold spheres of 500 nm diameter on GaAs substrate taken with a point source as shown in panel (b). In this case the imaged nano-objects do not present the coma-like shape as in panel (c).

where \( \Delta r \) is the outer-most zone width and \( m \) is the diffraction order of the ZP. Reaching this optimal highest spatial resolution of a FZP depends on many parameters. However, the experimental spatial resolution usually obtained by FZP systems is influenced by other extrinsic factors to be consider. Thus the experimental spatial resolution would be better calculated by [5].

\[ \delta = \sqrt{\delta_i^2 + \delta_g^2 + \delta_c^2} = \sqrt{\left(\frac{1.22 \Delta r}{m}\right)^2 + \left(\frac{\sigma q}{f}\right)^2 + \left(2r \Delta E / E\right)^2} \]  

where \( \delta_i \) is the intrinsic resolution of the ZP (equation 1), \( \delta_g \) is the demagnified source size with \( \sigma \) the source size and \( q \) and \( f \) the source FZP and focus distances, and \( \delta_c \) is the chromatic aberration being \( 2r \) the diameter of the FZP. Notice that the term \( \delta_g \), particularly takes into account the impact of a deformed and enlarged point source on the spatial resolution of the obtained image, (see figure 1b).

Figure 2c displays clearly the dramatic repercussions of a not well defined shape and size of the point source on the chemical and electronic images acquired with the ANTARES k-nanoscope. Effectively, rectangular topological insulator \( Sb_2Te_3 \) nanowires on a Si substrate are shown in the image with a coma-like shape due to a defective bad-defined point source. The image display the photoemission intensity of the Te 4d core level, however, instead to present the real shape of the nano-object, it show a fully distorted nanowire, that reproduces exactly the shape of the beam at the pinhole, (plane A in Fig. 1b). A similar image is obtained recording the PES
Figure 3. Focal distance optimization of the Fresnel Zone Plate  Panel (a) shows the image gold spheres on a GaAs substrate using a defocusing position of the FZP at the (C) plane. In the inset, the circular shape (black dashed line) of the nano-objects resembles the shape of the light spot at the plane (C), see Fig. 1b. Panel (b) displays the same image with the FZP located at the right focal distance at the plane (B).

intensity the Au 4f core level from severals spheres of Au on GaAs substrate in Fig. 2d. The coma-like shape of the gold nano-objects is missing because the point source has been optimized to a almost perfect spherical shape represented in Fig. 2b.

Moreover, the optimal spatial resolution is obtained following the standard procedure to determine the best (f) value, locating the FZP and the sample at the right distance. An example of the effect of the defocusing on an image is presented in figure 3. Both images have been obtained recording the PES intensity near Fermi level of a lithographic sample of gold spheres on a GaAs substrate. However, panel (a) show the distorted image obtained when the sample is placed in plane (C), while the high accurate image displayed in panel (b) is acquired when the sample is located at the optimal work distance, in plane (B).

3. Discussion
In summary, the ANTARES set-up has proved the feasibility of an innovative instrument where NanoARPES has been combined a well-focalized beam of X-rays together with a precise mechanical scanning system. The good reproducibility of the ANTARES k-nanoscope has been made possible by the optimization of sensible parameters of the BL optics.

4. Acknowledgments
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5. References
[1] Avila J and Asensio M C. 2014 Synchrotron Radiation News 27 24-30
[2] Bostwick, A. et al. 2012 Synchrotron Radiation News 25 19-25
[3] Jose, A. et al. 2013 Journal of Physics: Conference Series 425 192023
[4] O.E. Myers et al. 1951 Am. Jr. Physic. 19 357
[5] Yu, W et al. 199 Review of Scientic Instruments 70 2238