NanoXAS – The *in situ* Combination of Scanning Transmission X-ray and Scanning Probe Microscopy

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Abstract. NanoXAS is a novel x-ray microscope installed at the Swiss Light Source combining laterally resolved soft x-ray spectroscopy with scanning probe microscopy. We report on first *in situ* studies from thin polymer blend films and magnetic materials where topographic and spectroscopic contrast are used and show how complementary imaging modes provide new insight into many materials. In the future the scanning probe tip will be used to collect photoelectrons. By this we expect a spatial resolution in the few-nm range and unique information on surface and bulk properties of nano-materials.

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THE NanoXAS CONCEPT

The aim of the NanoXAS project is to combine the elemental and chemical sensitivity of soft x-ray spectroscopy with the high spatial resolution of scanning probe microscopy (SPM) [1]. NanoXAS is installed at a bending magnet beamline (spherical grating monochromator; photon energy range: 200 – 1800 eV; resolving power $E/\Delta E = 5.000$) of the Swiss Light Source. Figure 1 shows the setup schematically. The x-ray focusing is done upstream of the sample surface (left-hand side in Fig. 1). It consists of a Fresnel zone plate (FZP) and an order-sorting aperture (OSA) that combine to focus the light onto the sample ($\varnothing \approx 50$ nm). The FZP and OSA are mounted on piezo stepper motor stages that allow alignment with respect to the tip of the scanning probe microscope. The sample is mounted on a piezo scanner stage that allows raster scanning of the sample through the stationary x-ray focus, providing the lateral resolution. The scanning probe microscope (right-hand side of Fig. 1) is located downstream of the sample surface with the tip aligned coaxially to the x-ray beam.

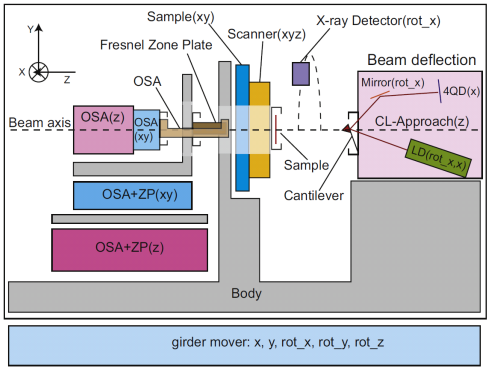

FIGURE 1. Sketch of the NanoXAS instrument. X-rays enter from the left and are focused onto the sample surface using a Fresnel zone plate (FZP) and an order sorting aperture (OSA). The sample is located on the scanner in the center and can be moved through the stationary x-ray. The sample surface points toward the tip of a scanning probe microscope. This tip is coaxial to the x-ray beam.

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As in a conventional scanning transmission x-ray microscope (STXM), the transmitted x-rays can be measured by a photodiode, providing a transmission image. In this case, the x-ray absorption provides elemental, chemical, and magnetic information.

The novel concept in NanoXAS is the coaxial arrangement of an SPM on the downstream side of the sample. The tip is fixed, and for image acquisition, only the sample is scanned. The SPM provides additional information by imaging the sample topography. Furthermore, the SPM tip can be used to collect the photoelectrons produced upon x-ray absorption in the sample. A detailed description can be found in [1].

IMPLEMENTATION

The instrument (see Fig. 2) was designed with the sample located in the center. It can be moved by 40 μm × 40 μm × 6 μm (x,y,z) using a linearized piezo scanner with closed-loop control and in addition by 5 mm × 5 mm (x,y) using piezo stepper motors. The x-ray illumination employing FZP focusing is located upstream. The SPM is located downstream of the sample and is realized as a beam deflection instrument with custom-built phase-locked loop (PLL) electronics. The positioning of all the elements in the instrument is done by 14 piezo stepper motors.

The instrument operates in ultrahigh vacuum (non-baked stainless steel vessel) at a typical pressure of $< 10^{-8}$ mbar. Samples and SPM-tips are transferred using a load lock.

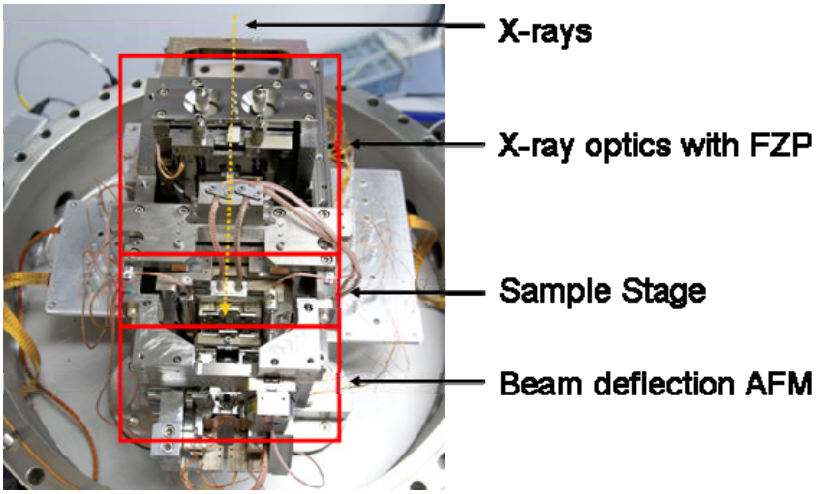


FIGURE 2. Photograph of the NanoXAS instrument with major components indicated. The inner diameter of the vacuum chamber is 600 mm.

NanoXAS TEST STUDIES

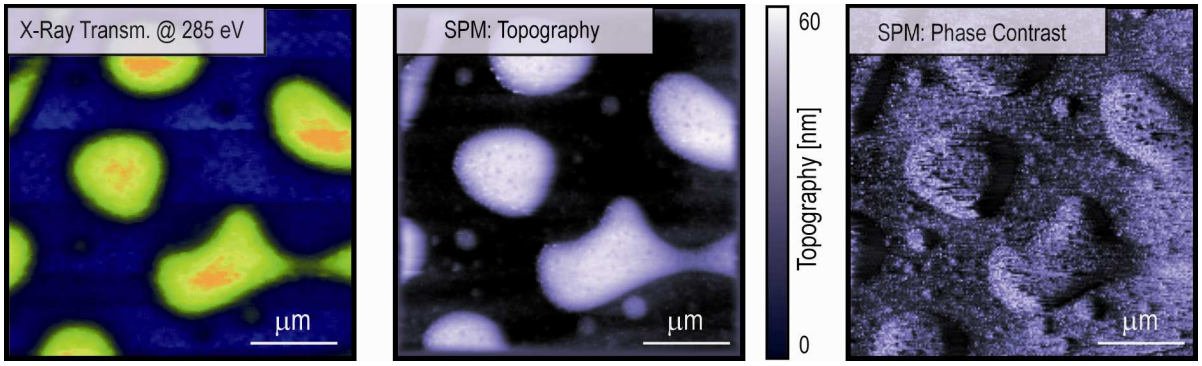
During the commissioning several prototype experiments were performed combining x-ray and scanning probe imaging at identical sample spots. The x-ray imaging used a photodiode to measure the transmitted x-ray intensity behind the sample. For SPM images, this photodiode is retracted. This change of imaging modes is computer controlled and takes only a few minutes.

Polymer Physics

Polymers and polymer blends are among the most intensely investigated systems in STXM microanalysis [2]. Polystyrene (PS) and poly-methylmetacrylate (PMMA) are immiscible polymers that phase separate in thermal equilibrium. Thin film PS/PMMA blends were prepared on Si_3N_4-membranes by spin casting from toluene solution. Annealing at 120°C initiated the phase separation.

Previous studies [2, 3] have shown that during phase separation some minor PMMA quantities are kinetically trapped within the PS matrix. Figure 3 shows three different images. Figure 3(a) is a false-color STXM image recorded at $h\nu = 285$ eV (close to the maximum absorption of PS), representing the lateral distribution and (not quantified) thickness of PS. In Fig. 3(b) an SPM topography image provides calibrated height information. The SPM

image confirms all major features of the STXM image, since the prominent morphological features are due to PS segregated areas. Figure 3(c) is the SPM phase contrast representing lateral sample-tip interaction or friction.



(a) (b) (c)

FIGURE 3. Polymer blend film imaged in the NanoXAS instrument showing (a) x-ray transmission, (b) SPM topography, and (c) SPM phase contrast. All images were taken *in situ* from the same sample spot (image size: 5 × 5 µm²).

Some smaller features are more clearly visible in the SPM topography (Fig. 3(b)) compared to the STXM image (Fig. 3(a)). The contrast becomes even more pronounced in the SPM phase contrast image (Fig. 3(c)).

These preliminary studies underscore the benefits of combined *in situ* studies of complex nanostructures. The chemical contrast in STXM can be directly correlated to the morphological/topographical features of surface-sensitive SPM. This is important since the 2D projection in STXM cannot directly be separated into surface or bulk-type information [4]. Conversely, SPM offers only limited chemical contrast. Imaging of the same sample position is easy in the NanoXAS instrument because of the coaxial design, while it is almost impossible to do when combining such experiments in different instruments *ex situ*.

Magnetic Imaging

NanoXAS is also a powerful tool to image magnetic nanostructures. X-ray illumination with circularly polarized photons [5] offers the possibility to use the x-ray magnetic circular dichroism (XMCD) contrast, i.e., the dependence of the absorption on the relative orientation of x-ray helicity and sample magnetization. On the other side, SPM with magnetic tips (MFM = magnetic force microscopy) measures the magnetic stray field of the sample, thus offering complementary information.

Images of an array of magnetic dots in a PdCo multilayer sample are shown in Fig. 4. We use (Ta(15A)/Pd(15A) $[Co(3A)/Pd(9A)]_8$Pd(9A)) multilayer where exchange coupling induces a magnetization perpendicular to the film. Figure 4(a) shows an x-ray absorption image taken at the Co L-edge ($h\nu$ = 778eV) in linear polarization. It provides

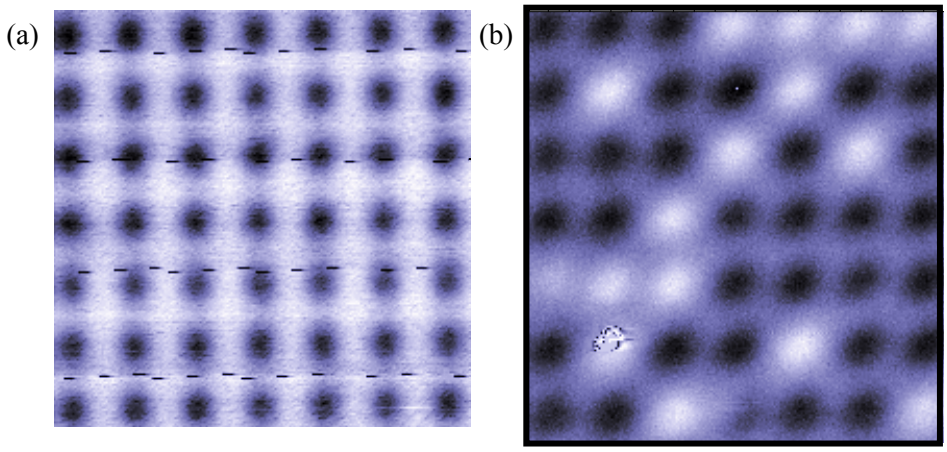


FIGURE 4. Array of magnetic CoPd multilayer dots: (a) STXM image taken at Co L3-edge (black streaks are artifacts due to non-corrected top-up injections); (b) MFM image. Images: 2 × 2 µm², dot Ø = 120 nm.

chemical information by imaging the Co in the CoPd dots. Figure 4(b) shows an MFM image using a vertically magnetized tip. Depending on whether the dots are magnetized up or down, the force between the sample and the tip is either attractive or repulsive. Thus this MFM images provides information on the magnetic orientation of the dots.

Future studies will include a more detailed spectroscopic analysis, i.e., the combination of XMCD for all involved elements with MFM.

OUTLOOK

Until now, STXM and SPM were used to obtain complementary information from the same sample spot. The next major step is the simultaneous use of both techniques. We plan to detect the secondary electrons produced by the x-ray illumination using a conductive SPM tip. Using a coaxially shielded SPM tip, only the electrons within a small radius around the tip center are accepted thus improving the spatial resolution below the diameter of the illuminated spot [1]. Our ultimate goal is to push the spatial resolution below 5 nm.

Different additional imaging modes are under development. The availability of ultra-high vacuum allows the detection of secondary electrons. Total electron yield measurements can be realized by measuring the drain current or by direct detection of the secondary electrons using a channeltron. In the latter case, surface sensitivity in STXM analysis can greatly be enhanced [4, 6]. SPM also allows us to measure local conductance maps or Kelvin probe force microscopy.

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