NanoXAS, a novel concept for high resolution microscopy

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Abstract. We currently develop a novel type of scanning x-ray microscope. This instrument will combine the chemical specificity of x-ray absorption spectroscopy with the very high spatial resolution of scanning probe microscopy. In a fundamentally new instrumental approach, the instrument can be used as a conventional scanning transmission x-ray microscope (STXM), as a conventional scanning probe microscope (SPM) or in a mode combining these two techniques. In the latter case, the sample is placed in the focus generated by the fresnel zone plate of the STXM. The SPM-tip placed downstream acts as a local detector of the emitted photoelectrons. Simulations and experiments have shown that the use of shielded SPM-tips is crucial to obtain a strongly increased chemical resolution. In contrast to similar projects underway at other synchrotrons we use a coaxial geometry. This should greatly enhance the flux density and reduce background signals caused by straylight illuminating the tip.

1. Introduction

In materials science there are two different techniques dominating the field: X-ray microscopy and scanning probe microscopy (SPM). While x-ray microscopy provides chemical sensitivity, but is currently limited to $x \sim 15\text{nm}$ resolution [1], SPM provides sub-nm resolution but with no or only little chemical information. Some combination of the two techniques would be advantageous if it could combine the spatial resolution of SPM with chemical sensitivity of X-ray microscopy. The basic principle of combining x-ray absorption spectroscopy (XAS) and scanning tunneling microscopy (STM) has been previously demonstrated by Eguchi et al. [2] and Saito et al. [3], who reported element specific images acquired by an STM tip rastered over a x-ray illuminated surface. Thus, acquiring two-dimensional chemical images as well as localized spectral analysis through a STM tip is possible, although the measured photocurrents are weak (pA) compared to the tunneling currents (nA). For these experiments the sample was illuminated by the X-ray beam at grazing incidence, which results in a very large area of the sample as well as the tip being illuminated to create a very large background signal. This approach has the further difficulty of possible intermixing of the distance feedback and the photoelectron signal. S. Suzuki et al. [4] avoided signal intermixing by combining XAS with non-contact atomic force microscopy (NC-AFM) [5]. They detected a peak in the measured frequency shift signal of the oscillating cantilever when the photon energy was scanned over the absorption edge of the underlying...
We propose a new technical approach for combining XAS with SPM: A conventional scanning transmission x-ray microscope (STXM) setup using a fresnel zone plate (FZP) to focus the x-ray beam to a spot size < 50 nm in diameter and thus increasing the emitted photoelectron density, is one part of the instrument. On the other side of the sample, a coaxially insulated cantilever tip is placed in the center of the focused beam in order to locally collect the emitted photoelectrons. Note that this arrangement, illustrated in Fig. 1 (a), requires that the tip be downstream of the sample and thus the sample is illuminated through the back-side. The use of the scanning force microscopy (SFM) instead of an STM-technique has the advantage that the tip-sample distance can be controlled by the resonance frequency of the cantilever, which is clearly separated from the photocurrent signal of interest. Furthermore, the instrument can also be used in normal STXM- or SFM-mode as complementary techniques. The instrument, named NanoXAS, will be operational at a dedicated beamline of the Swiss Light Source (SLS) by July 2009.

2. Description of Instrument and Beamline
The NanoXAS instrument is designed to combine three experimental techniques: STXM, SFM and their combination. NanoXAS will be built in a UHV-chamber, attached to bend magnet beamline X07DB. The optical layout of the beamline very similar to the PolLux [6] beamline (X07DA) of the SLS, but will have an extended energy range of 200-1600 eV. A load-lock chamber attached to the main UHV-chamber and two manipulators will allow for fast sample and cantilever exchange under UHV-conditions. The principle of the NanoXAS instrument is shown in Fig. 1 (a). The FZP focuses the soft x-ray light to a diffraction limited spot. The focal length of the FZP depends on the photon energy. Higher diffraction orders are filtered out using an order sorting aperture (OSA), located between the FZP and the sample. The OSA is positioned so that it provides maximum rejection of higher diffraction orders and maximum transmission of first order light through its aperture. The distance between OSA and the sample is typically less than 1 mm, depending on the photon energy. The sample is deposited on a semitransparent SiN-membrane or mounted on a transmission electron microscope grid. To allow conventional STXM experiments a large (ø ~ 1 mm) photodiode detector can measure the transmitted x-ray intensity. If the photodiode is retracted, the SPM-tip can approach the sample from the downstream side where it collects the emitted photoelectrons. This tip current, $I_{\text{tip}}$, is preamplified using a low noise I/V converter. In addition, a lock-in amplifier can be

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Figure 1. Sketch of the NanoXAS instrument (a) and schematic view of coaxial SPM-tip located in the focus of the zone plate with arrows indicating the photoelectrons
used to filter out unwanted background as caused, for example, by direct illumination of the tip. For lock-in amplification, the incoming x-ray beam can be chopped at about 1 kHz and the monochromator allows modulation of the photon energy by $\sim 50$ eV/s.

Imaging is accomplished by scanning the sample, mounted on a fine- (closed-loop scanner $40 \times 40 \times 5 \mu$m) and coarse-stage (positioner), across the x-ray focus. The positioner allows positioning the relevant sample region in the x-ray beam. In order to control the tip-sample distance, $z_{ts}$, dynamic scanning force microscopy operation modes will be used. In the constant frequency shift mode, $z_{ts}$ can be controlled to fractions of nanometers whereas in the constant height mode the average $z_{ts}$ is kept constant and can be adjusted to about 5-50 nm, depending on the sample surface.

3. Coaxial Cantilever Design and approximation of measured Tip current

The scientific and technological challenge of the NanoXAS project lies in controlling the diameter of the probed area, $D_{probe}$, from which the SPM-tip collects photoelectrons and measures them as a current, $I_{tip}$. Simulations performed by Chiu et al. [7] discuss the resolution limits of insulated STM-tips irradiated by x-ray light. They define $D_{prob}$ as the diameter of a circular region on the sample surface within which emitted electrons contribute to 90% of $I_{tip}$. Obviously, $D_{prob}$ will limit the spatial resolution of the instrument $\Delta x \sim D_{prob}$.

Since photo electrons are emitted in the entire illuminated area $A_{illum}$ having a diameter $D_{illum} \sim 40$ nm, the challenge is in preventing electrons produced far from the tip from reaching the tip and contributing to the signal. This requires a small tip radius, $r$, a very effective shielding of the tip [8, 9, 10], a small tip-sample distance ($z_{ts}$) as well as an adequate bias voltage on the tip, $U_{tip}$, and the shield, $U_{shield}$. Fig. 1 (b) shows the planned layout of a coaxial SPM-tip. It consists of an inner conductor (red) made of doped Silicon, an insulating SiO$_x$ layer and a thin conducting PtC shield layer of several nm each. For the fabrication of such tips we plan to use high aspect ratio silicon cantilevers, produce a thin oxide layer by thermal oxidation and then to deposit a thin, smooth PtC layer by e-beam evaporation. Finally, the shield and the insulation layers are removed from the tip apex by focused ion beam (FIB).

In order to get an idea about the size of $I_{tip}$ that can be expected, we first measured the photoelectron density, i.e. the number of emitted photoelectrons per area $I_{sample}/A_{illum}$, where $I_{sample}$ is the drain current of the sample and $A_{illum}$ is the total illuminated sample area. This is experimentally done by measuring the drain current of an illuminated cobalt test sample using the STXM [6] at the PolLux beamline of the SLS. For a focus diameter of $\sim 35$ nm the measured drain current at the Co L$_3$ absorption edge (778.4 eV) was about 3 pA. This results in a photocurrent density of about 4000 A/m$^2$, so compared to a typical photocurrent density of a non-focused undulator source (U5 at the NSRRC [7]) of about 0.7 A/m$^2$, we gain a factor of more than 1000.

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