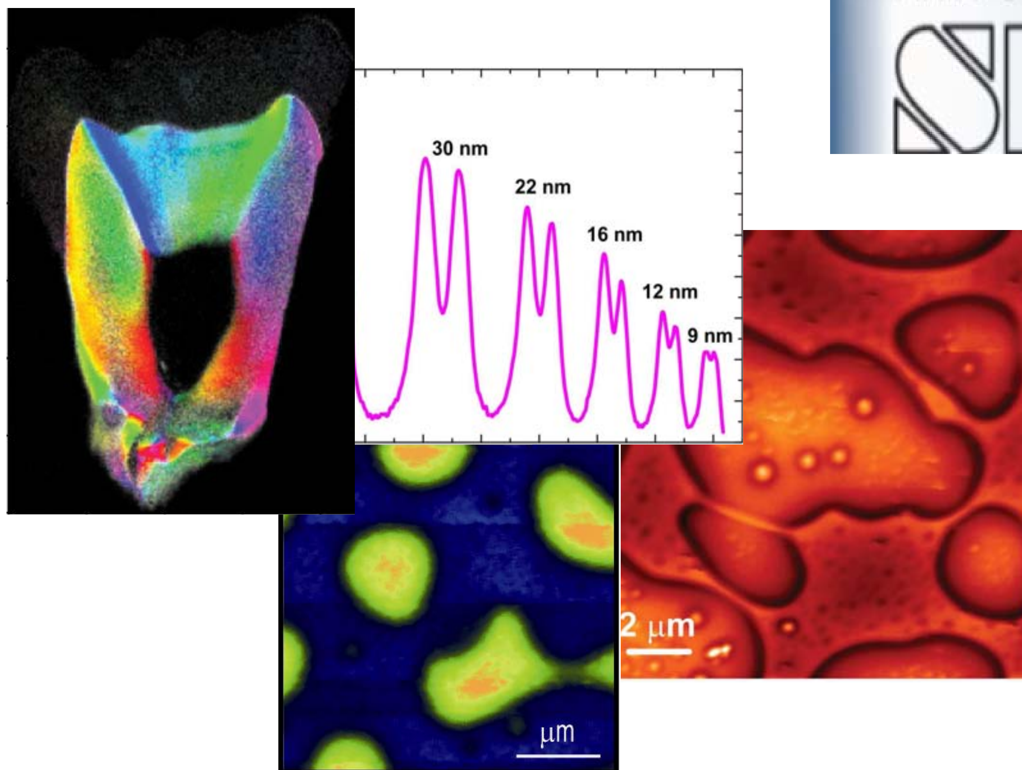

SWISS LIGHT SOURCE

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SLS Symposium on

Scanning X-ray microscopy

Tuesday, May 4, 2010

10:00 to 12:15, WBGB/019

10:00 NanoXAS – an innovative tool combining Scanning Probe and X-Ray Microscopy
I. Schmid, J. Raabe, Ch. Quitmann and H.J. Hug

10:30 Simultaneous surface and bulk imaging of polymer blends with xray spectromicroscopy
B. Watts and C.R. McNeil

11:00 Coffee

11:15 Scanning small-angle x-ray scattering on human tissues
H. Deyhle, G. Schulz, S. Mushkolaj, B. Mueller and O. Bunk

11:45 Nanofabrication Methods for High-Resolution Scanning Transmission X-ray Microscopy
J. Vila-Comamala, S. Gorelick, V. A. Guzenko, E. Färm, C. M. Kewish, A. Diaz, J. Raabe, M. Ritala, A. Menzel, O.Bunk and C. David

NanoXAS – an innovative Tool combining Scanning Probe and X-Ray Microscopy

I. Schmid[a)], J. Raabe[a)], Ch. Quitmann[a)], and H.J. Hug [b,c)]

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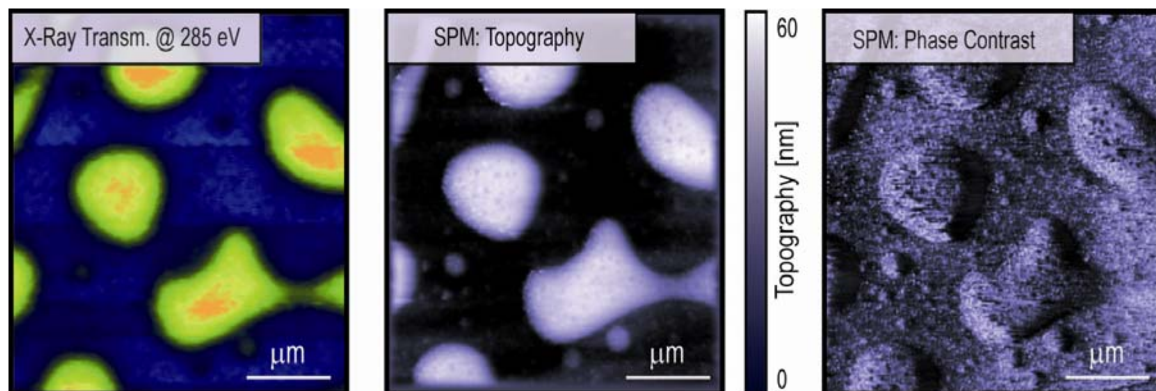
In materials science there are two different techniques dominating the field: X-ray microscopy and scanning probe microscopy (SPM). While x-ray microscopy provides chemical sensitivity, but is currently limited to about 15nm resolution, SPM provides sub-nm resolution but with no or only little chemical information. Combining the two techniques, i.e. the spatial resolution of SPM with the chemical sensitivity of X-ray microscopy, would thus be very advantageous.

We show 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. The instrument, named NanoXAS, is currently being commissioned and first results are presented.



NanoXAS allows combination of STXM (left) SPM topography (center) and SPM phase contrast in-situ. This is unique and a "first".Different polymers can be uniquely identified due to their tabulated absorption spectra. STXM is mostly bulk sensitive. SPM provides high resolution (<40nm given by sample structures) and surface sensitive information.The small defects in the PMMA blobs are only poorly visible in STXM because of their small volume. This will change when we detect photoelectrons either with the tip or with a channeltron.

Simultaneous surface and bulk imaging of polymer blends with x-ray spectromicroscopy

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We demonstrate the utility of soft x-ray spectromicroscopy to simultaneously image the surface and bulk composition of polymer blend thin films. In addition to conventional scanning transmission x-ray microscopy that employs a scintillator and photomultiplier tube to measure the transmitted x-ray flux, channeltron detection of near-surface photoelectrons is employed to provide information of the composition of the first few nanometers of the film. Laterally phase-separated blends of two polyfluorene co-polymers, poly(9,9'-dioctylfluorene-co-bis(N,N'-(4,butylphenyl))-bis(N,N'-phenyl-1,4-phenylene)diamine) (PFB) and poly(9,9'-dioctylfluorene-co-benzothiadiazole) (F8BT), are studied, with the structure of both wetting and capping layers clearly imaged. This new information provides insight into the connectivity of bulk and surface structures that is of particular relevance to the operation of such blends in optoelectronic devices. In particular we show that the F8BT droplets enclosed in the PFB-rich phase penetrate the PFB-wetting layer connecting the top and bottom surfaces of the film. As these films are incorporated into devices by sandwiching between two electrodes, being able to determine which parts of the film are connected to which electrodes is important in order to understand device operation and to develop morphologically correct device models.

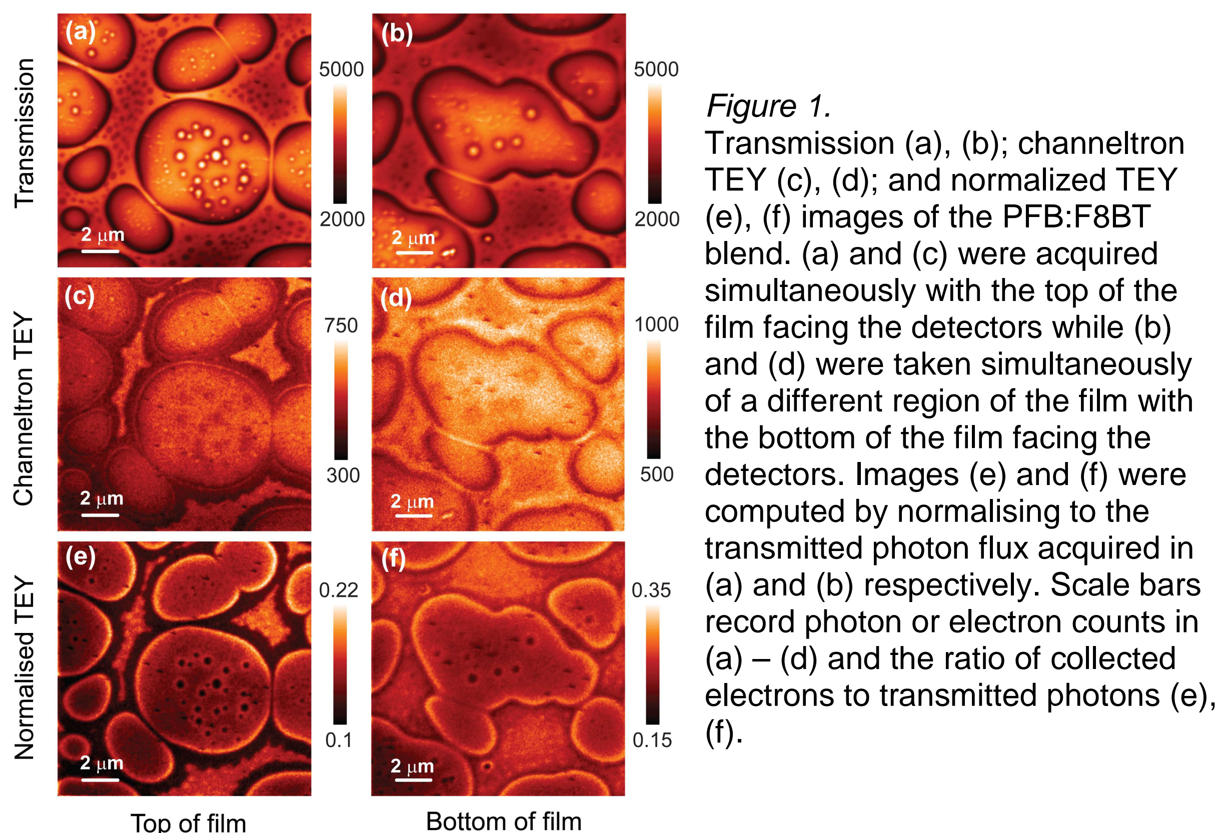

Figure 1.
Transmission (a), (b); channeltron TEY (c), (d); and normalized TEY (e), (f) images of the PFB:F8BT blend. (a) and (c) were acquired simultaneously with the top of the film facing the detectors while (b) and (d) were taken simultaneously of a different region of the film with the bottom of the film facing the detectors. Images (e) and (f) were computed by normalising to the transmitted photon flux acquired in (a) and (b) respectively. Scale bars record photon or electron counts in (a) – (d) and the ratio of collected electrons to transmitted photons (e), (f).

Scanning small-angle x-ray scattering on human tissues

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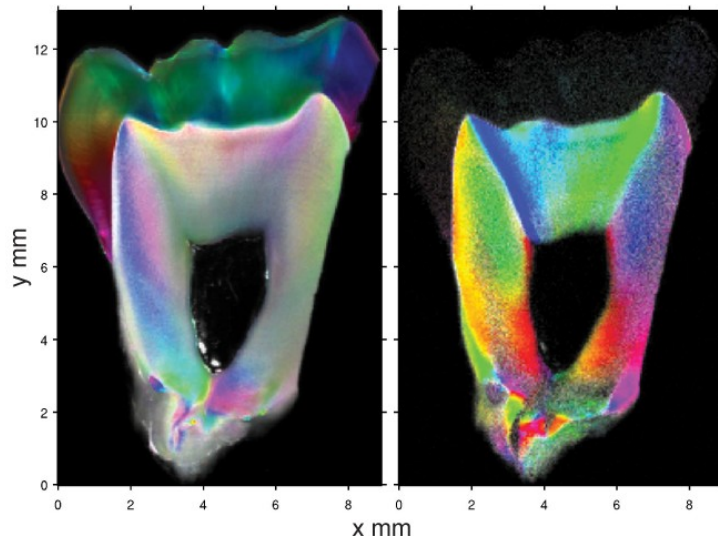
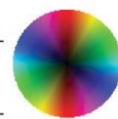
For the full characterization of a tissue, information on all length scales, from macroscopic to the molecular level are needed. Collecting structural information in the nanometer range over extended areas is too time consuming to be feasible with state of the art techniques such as electron microscopy. and the specimens are typically far away from physiological conditions. Scanning small angle x-ray scattering (SAXS) reveals a wealth of information on the texture of biological samples in the nanometer range over extended areas within reasonable time and without the need for complex sample preparation. The technique will be explained briefly followed by examples where scanning SAXS has been applied to different human tissues like thalamus, urea and human teeth with the focus on the latter.

Teeth are composed of anisotropic materials, namely dentin and enamel. Enamel is hard and brittle, composed mainly of densely packed calcium phosphate organized in prisms or rods, with few organic components. Dentin while still composed mainly of inorganic calcium phosphates, is interwoven by collagen I, making it tougher and allowing to absorb or redistribute stress. The combination of these two components makes a highly durable structure, which lasts a lifetime under extreme mechanical and chemical conditions. Especially the dentin-enamel junction (DEJ), the interface between dentin and enamel acts as a crack barrier, preventing cracks formed in the brittle enamel to propagate through the entire tooth.

SAXS reveals a wealth of information on the texture of dentin and enamel in the nanometer range. Nanostructures in the enamel yield scattering signals approximately parallel to the DEJ, while the scattering of inorganic substance in dentin is oriented mostly perpendicular to the junction. The characteristic scattering signal of collagen at 67 nm allows to extract the collagen contributions, revealing the parallel orientation of the organic collagen to the dentin crystallites.





Left, scattering signal between 61.1 and 68.8 nm of a 400 µm thin tooth slice recorded in scanning SAXS. The color wheel gives the orientation of the scattering signal. The saturation of the colors codes the oriented scattering intensity while the color intensity codes the total scattering intensity. On the right, the extracted collagen signal from the same scan.

Nanofabrication Methods for High-Resolution Scanning Transmission X-ray Microscopy

J. Vila-Comamala[1], S. Gorelick[1], V. A. Guzenko[1], E. Färm[2], C. M. Kewish[1], A. Diaz[1], J. Raabe[1], M. Ritala[2], A. Menzel[1], O.Bunk[1], C. David[1]

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Current synchrotron-radiation-based techniques demand for extremely small X-ray probes, typically no bigger than a few tens of nanometers. Nanofabrication methods are crucial to improve the performance of X-ray microscopes that use X-ray diffractive optical elements as focusing devices. We demonstrate the highest spatial resolution ever reported in scanning transmission X-ray microscopy (STXM) to date. For the first time in X-ray microscopy, features below 10 nm in width were resolved in the soft X-ray regime (1.2 keV) and 15 nm lines and spaces were visible at multi-keV photon energies (6.2 keV). These achievements were accomplished by using line-double Fresnel zone plates (FZP). The lenses were fabricated by combining electron-beam lithography with atomic layer deposition [1]. In the soft X-ray regime, the spatial resolution tests were performed by acquiring transmission images of line pair structures of a GaAs/AlGaAs heterostructure. A zone-doubled Fresnel zone plate with 100 µm diameter and outermost zone width of 12.5 nm was used. Figure 1 shows the STXM image of the sample containing line widths ranging from 40 to 9 nm. The set of three lines of 9 nm is resolved [2]. During the STXM experiments at multi-keV photon energies, the full divergent radiation cone was sequentially recorded by a pixel fast framing detector at each scanning position. These data were processed to obtain the transmission, dark-field, differential phase contrast and the integrated phase signals for every sample. A zone-doubled Fresnel zone plate with 100 µm diameter and outermost zone width of 15 nm was used. The STXM image of test structures shown in figure 2 demonstrates the extreme resolving power. In addition, high spatial resolution STXM images of several biological specimens have been acquired in transmission, dark-field and differential phase contrast modes.

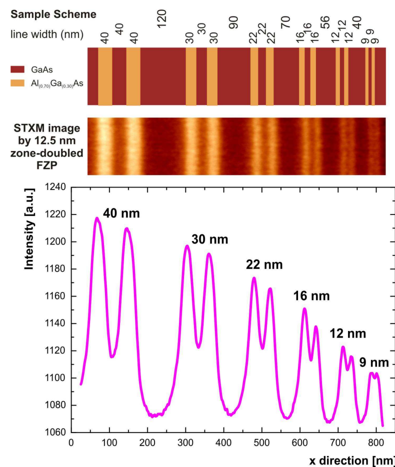

Figure 1: STXM image of GaAs/AlGaAs hetero-structure acquired at 1.2 keV photon energy by zone-doubled FZP with 12.5 nm outermost zone width. The set of three lines of 9 nm is resolved.

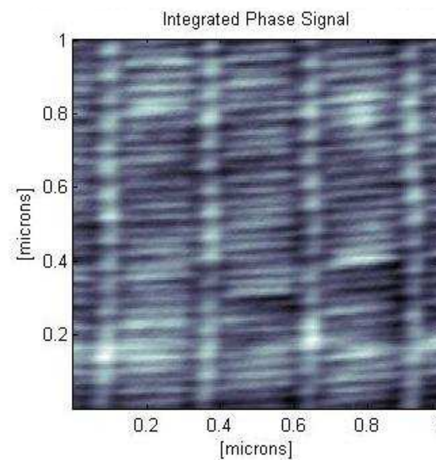

Figure 2: STXM image of 15 nm lines and spaces resolved at 6.2 keV photon energy. The data were acquired by a zone-doubled FZP outermost zone width of 15 nm.

References:

[1] K. Jefimovs, J. Vila-Comamala, T. Pilvi, J. Raabe, M. Ritala, C. David. Phys. Rev. Lett. 99, 264801 (2007)

[2] J. Vila-Comamala, K. Jefimovs, J. Raabe, T. Pilvi, R. H. Fink, M. Senoner, A. Maassdorf, M. Ritala, C. David. Ultramicroscopy 109(11), 1360-1364 (2009)