

Experiment title: High spatial resolution element mapping with a transmission X-ray microscope

Experiment number:

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Report:

Up to now mainly scanning electron microscopes (SEMs) are used to excite X-ray fluorescence of atoms by focusing accelerated electrons on the sample. The interesting elements are detected by their energy specific X-ray emission. X-ray fluorescence of atoms can also be excited with X-rays. Using quasi monochromatic X-rays instead of electrons has the advantage of a higher selectivity and therefore the dose applied to the sample is lower. Another advantage is that hydrated and much thicker samples can be studied, because X-rays can penetrate into larger depth. Usually scanning transmission X-ray microscopes are used to collect data of atomic species distributions in samples. In these microscopes the sample is scanned pixel per pixel in the small diffraction limited focus generated by a zone plate objective. This procedure combines relatively low dose applied to the sample, high sensitivity and gives the element distribution of many different elements at the same time. However, the main disadvantage of this method is the low signal combined with the sequential image formation caused by the scanning process, which leads to very long exposure times of up to hours and makes it difficult to obtain images with large numbers of resolution elements or pixels.

Alternatively, a fullfield transmission X-ray microscope could be used to detect many interesting elements (e.g.,Mg, K, Ca, P, S) by measuring the changes of the X-ray absorption at their element specific inner-shell absorption edges. In this case two images of the sample have to be taken before and behind the inner-shell absorption edge of the interesting element. The absorption method of the TXM has the main advantage that no mechanical movement of

the sample or the X-ray objective is required, which also avoids loss of spatial resolution caused by vibrations and non-linear differential posistioning - unequal - step sizes during the scan-movement in the STXM. With a TXM high-resolution, short exposure times and large numbers of pixels (typically 1024 x 1024 pixels on the CCD chip) would be obtained.

However, in order to apply this method in the TXM, the strong zero-order radiation has to be drastically reduced so that the image contrast is increased and better adapted to the limited dynamical range of the X-ray CCD camera in the TXM. This can be done in the Fourier plane by using a strongly absorbing ring similar in shape to a phase-plate in the phase-contrast microscope. However, its phase-shift is not relevant, but only its absorption, which determines the image contrast. As already shown by theory the zero-order radiation can drastically be reduced without significant change of the required photon density or dose.

To verify the theory we performed experiments using the TXM installed at ID21 at the ESRF. At first we installed an absorption plate manufactured from 3 μ m thick gold in the back-focal plane of the TXM and adjusted it in a way that the zero order radiation of the object was reduced in intensity. Indeed we could observe an increased image contrast (Fig.1). For this purpose we used the direct beam without hollow cone illumination to illuminate the object. The next step will be in the future to adjust the rotating condenser in order to realize a hollow cone illumination of the object, which is required to avoid direct light in the image plane and makes it possible to transmit the zero order radiation through an annular phase plate in the back-focal plane. Such an X-ray optical setup would avoid a significant background, which has disturbed the image contrast when using the direct beam.

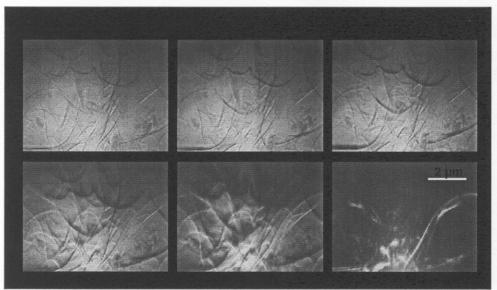


Fig.1: Image sequence of a 20µm thick structured polymer, the image in the upper left is bright field absorption image without phase zone plate in the back-Fourier plane, in the following images the phase plate is subsequently moved into the beam