Comparison of grazing incidence small angle X-ray scattering of a titania thin film sponge structure at BW4 (DORIS III) and P03 (PETRA III)

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Grazing incidence small angle X-ray scattering (GISAXS) is a powerful tool to investigate structures of molecular up to mesoscopic scales in thin film geometries [1].

GISAXS measurements have been performed at the HASYLAB beamlines BW4 at the DORIS III storage ring and at P03 at the PETRA III storage ring. At the beamline BW4 synchrotron radiation with a wavelength of 0.1381 nm, a beam size of 23×36 μm^2 (vertical × horizontal direction) and an incident angle of 0.35° on the surface of the films was used for the measurements. The sample-to-detector distance was set to 2 m. The scattered signal was detected with a two-dimensional MarCCD detector with 2048×2048 pixels of a pixel size of 79.1×79.1 μm^2 and an active area of 165 mm in diameter with a read-out noise of 10 counts/pixel. At the beamline P03 synchrotron radiation with a wavelength of 0.0969 nm, a beam size of 20×40 μm^2 (vertical × horizontal direction) and an incident angle of 0.28° on the surface of the films was used for the measurements. The incident angle was adjusted so as to detect the specular reflection approximately at the same q_z-value for both measurements. The sample-to-detector distance at P03 was set to 2.5 m. The scattered signal was detected with a two-dimensional Pilatus 300k detector with 487×619 pixels of a pixel size of 172×172 μm^2 and an active area of 83.8×106.5 mm^2 with no read-out noise.

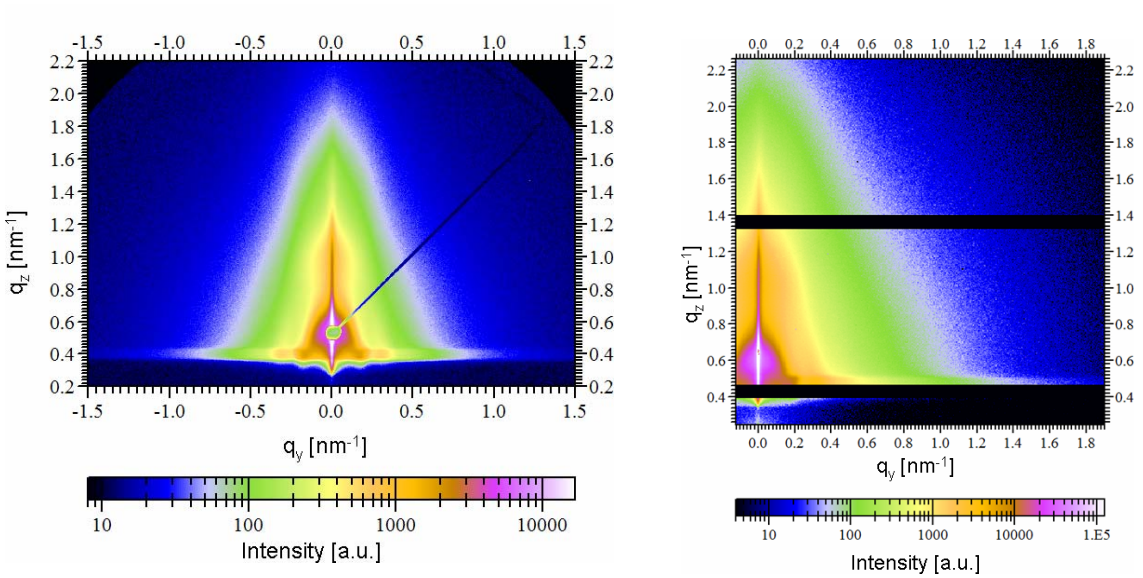

Figure 1: *Two-dimensional GISAXS scattering patterns of a titania sponge structure as measured at the DORIS III beamline BW4 (left) and at the PETRA III beamline P03 (right).*

The investigated sample consists of a titania sponge structure which was nanostructured with a combination of block copolymer based sol-gel templating and micro-fluidics [2]. The titania thin film has a thickness of about 215 nm on top of a glass substrate.

Figure 1 shows a GISAXS scattering pattern of the titania sponge structure measured at BW4 for 300 s in multiread mode, featuring a beamstop to block the high intensity of the specular reflection, and a GISAXS scattering pattern of the same titania structure measured at P03 for 0.3 s. The asymmetric setup of the Pilatus 300k detector in the measurements at P03 was chosen to probe approximately the same q-range as in the measurements at BW4 with the MarCCD detector. Horizontal and vertical cuts of the GISAXS data are shown in figure 2. In the horizontal cuts the same structure information can be obtained, where the difference in noise of the two detectors is visible in the different slopes for large q-values. Both cuts reveal the same structure factor peak with the same higher orders. The position of the specular reflection in the vertical cuts reveals a slight mismatch of the q_z values between both set-ups.

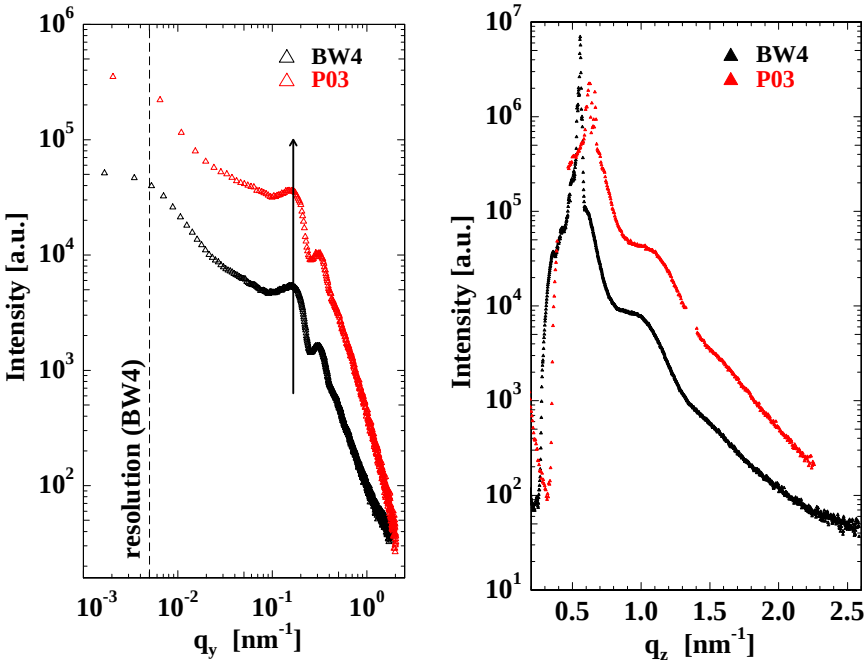

Figure 2: *Horizontal cuts at the same q_z-position for both sets of GISAXS data (left) and vertical cuts of the same GISAXS data for q_y=0 (right). The structure factor peak, indicated by the arrow in the horizontal cuts, corresponds to a size of about 38 nm. The cuts are not shifted along the intensity axes, the difference in intensity is due to the higher flux at P03 and the different pixel sizes of the detectors.*

The most interesting detail in the comparison of the GISAXS data obtained at the two different beamlines is the huge difference in intensity. The intensity, as detected at the Yoneda peak position, is higher in the P03 data by a factor of approximately 6.9. Normalized by the different pixel area this leads to a factor of 1.5 in the intensity. The different measurement time of 300 s in multiread mode, which is effectively a measurement time of 150s, at BW4 and a measurement time of 0.3 s at P03 leads to an additional factor of 500. In total the gain of intensity per time and per detector area for the titania sample amounts to a factor of about 750, neglecting the small differences caused by the small deviation in q_z. In conclusion GISAXS measurements at the two different beamlines BW4 and P03 yield the same structural information, with a significantly higher intensity per detector area and time at the new beamline P03 at the third generation storage ring PETRA III.

References

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