Scattering experiments at the BW4

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The BW4 is a dedicated SAXS beamline, which has the capabilities for normal SAXS as well as für advanced techniques like USAXS and GISAXS. The real space resolutions are $2\,\mathrm{nm}$ to $200\,\mathrm{nm}$ in SAXS and $1\,\mathrm{nm}$ to $400\,\mathrm{nm}$ in GISAXS. With sample to detector distances of up to $13.5\,\mathrm{m}$ the upper limits for USAXS and GIUSAXS are $1\,\mu\mathrm{m}$ and $13\,\mu\mathrm{m}$, respectively. There is also a moderate micro-focus option available which is produced by a Beryllium-compound refractive lens (Be-CRL). With this a beam size down to $32 \times 17\,\mu\mathrm{m}^2$ (H×V) possible. Special sample environments provided are heating stages for transmission and grazing incident measurements as well as a stretching device. [1]
To show the feasibility of such optics there was a temporary setup was a USAXS measurement with a Be-CRL as focusing optic. This was done without mirrors and gave a total flux of $5 \times 10^{11}\ \mathrm{photons}/\mathrm{s}$ at sample position. Figure 1 shows the scattering pattern of a collagen fibre with this setup at a sample detector distance of $12.6\,\mathrm{m}$.

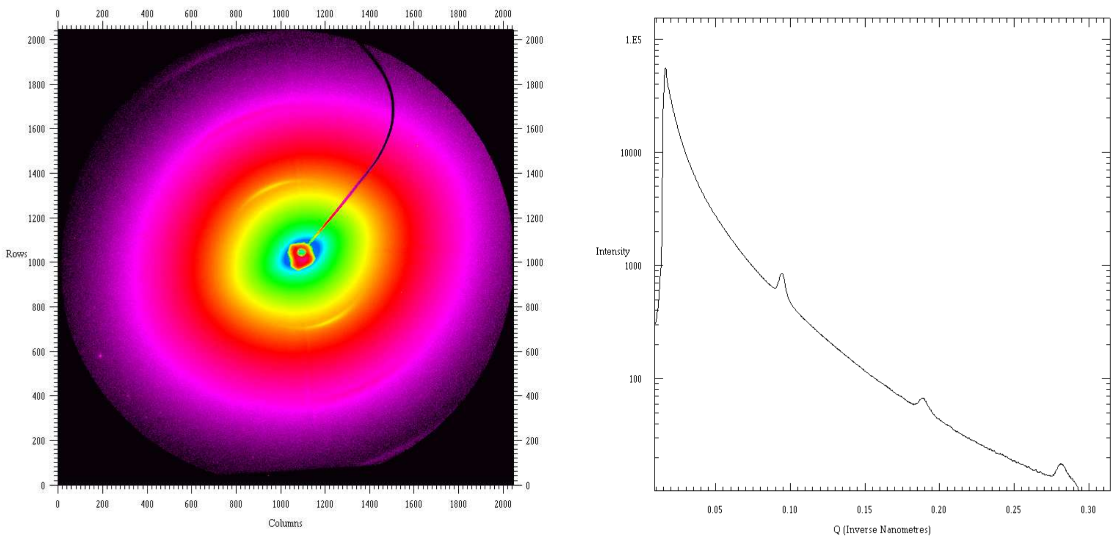

Figure 1: Scattering pattern(left) and averaged scattering curve(right) of a collagen fibre measured with USAXS using a BeCRL as focusing optic.

Another experiment conducted at the BW4 was an in-situ GISAXS measurement of a colloid solution unter shear stress. A $13\,\mathrm{wt.\%}$ solution of a poly-(isoprene-block-ethylene oxide) in water forming a FCC lattice was investigated. The block degrees of polymerization of the polyisoprene and the polyethylene oxide were 55 and 170, respectively. [2] The shear stress was applied by a stress-controlled Bohlin CVO rheometer in a plate-plate-geometry with a diameter of $20\,\mathrm{mm}$ and a gap of $1\,\mathrm{mm}$. We investigated the interface layer of the solution with the rotor of the shear geometry. The measurements were conducted at different temperatures. The figure 2 shows the result of the measurements at a temperature of $T = 15\,^{\circ}$. This is representative for all temperatures.
We found that the layer thickness of the metal-solution interface increases with the first application of shear. After that the increase of the shear rate doesn't change the thickness of the layer anymore. From this we conclude that the micelles deposited at the metal surface get detached by the application of shear stress and then stay at a constant distance to the surface. This also leads to the conclusion that the first micellar layer slips under shear.

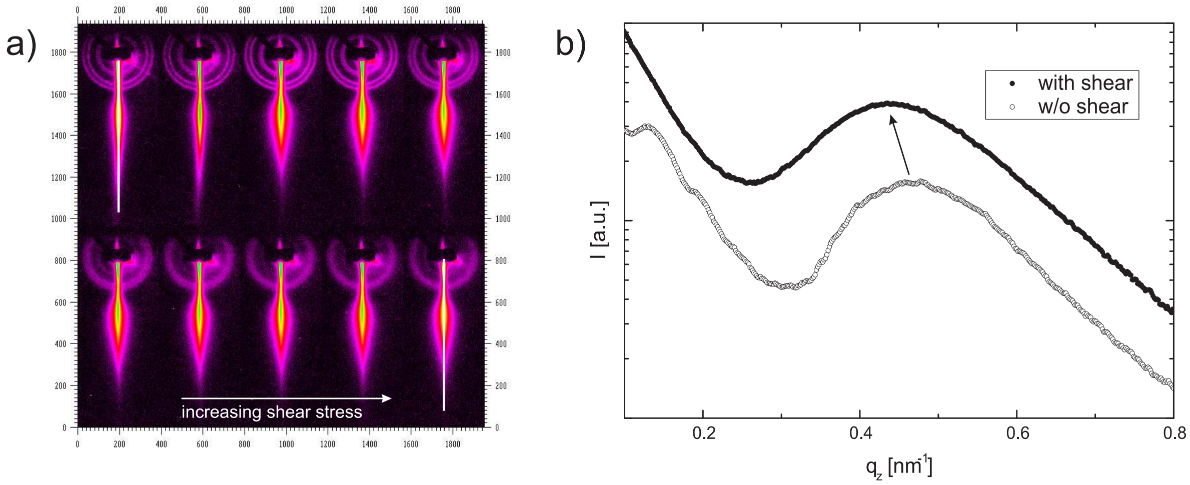

Figure 2: Scattering patterns at the rotor wit increasing shear (a). The white lines indicate the detector cuts without shear stress and at $\tau = 120\,\mathrm{Pa}$ (b).

References

[1] S.V. Roth et al., Rev. Sci. Instr. 77, 085106 (2006)
[2] A. Timmann, doctoral thesis, Görich und Weiershäuser, Marburg, 2006