In-situ lyotropic phase mapping combining microfluidics and microfocus SAXS at MiNaXs Beamline/Petra III

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Due to its high-resolution scanning capacity, analytical methods using X-ray or synchrotron radiation are excellently suitable for the characterization of nanoscale materials. Particle sizes and shapes, as well as structural changes can be easily investigated.[1] In order to study kinetic processes the combination of state-of-the-art X-ray scattering methods and microfluidic setups is very promising. Since the temporal evolution of a kinetic process is mapped onto different positions along the microchannel, time resolution is given by the spatial resolution of the detection method. Furthermore, using microfluidics it is possible to obtain well-defined nanoscale structures in a controlled way because of a laminar flow conditions.[2]

Here we present a first study on the kinetic process of *in-situ* lyotropic phase formation of Poly-(dimethylsiloxane-*b*-ethylene oxide) (PDMS-*b*-PEO) in a microchannel. We use X-ray transmittant microfluidic devices consisting of a UV curable adhesive (*Norland optical adhesive 81 (NOA81)*) fabricated by soft lithographic procedures.[3] The progression of the structural changes can be observed on different positions along the microchannel by micro-focus small angle X-ray scattering (SAXS) and evaluated using *Scatter*[4].
In our experiments, a solution of PDMS-*b*-PEO in *iso*-propanole (75% v/v) is hydrodynamically focused from both sides with water, each of them at a constant flow speed of 50 μl/h using high-precision syringe pumps. The spatial dimensions of the channel are 150 μm in width and 100 μm in height. We use devices completely composed of NOA81 and a lithographic procedure which allows the fabrication of thin (ca. 300 μm in beam path) devices.

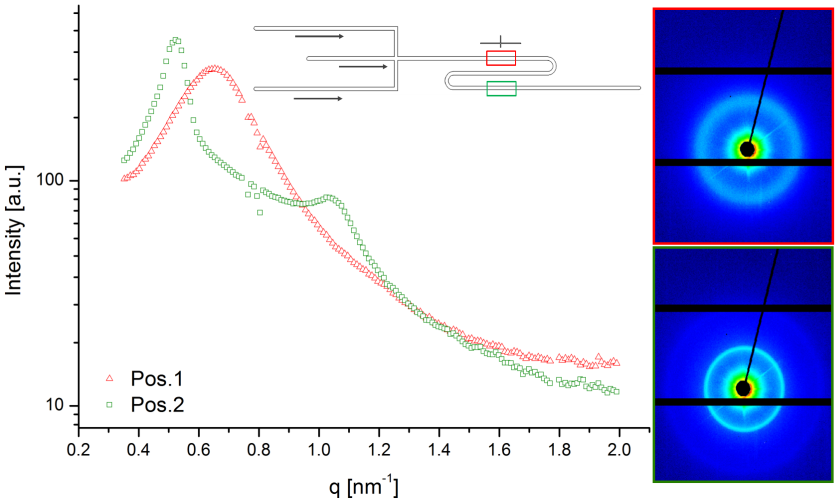

Figure 1: scattering curves and corresponding scattering patterns (*top:* Pos.1, *down:* Pos.2) measured at two positions of the microchannel with a scheme of the used microchannel (arrows indicate liquid flow)

The synchrotron beam with a size of 13x22 μm (vertical x horizontal direction) operates at a wavelength of 0.95 Å and was used to scan the channel and to investigate the lyotropic phase along and across the microchannel. The scattered radiation was detected by a Pilatus 300 K detector at a distance of 2.00 m. Its 487 x 619 pixels have a size of 172 x 172 μm^2 and are distributed on an area of 83.8 x 106.5 mm^2.

The scattering patterns corresponding to two positions along the microchannel are shown in Figure 1. The first position was arranged 8 mm (equivalent to ca. 6 s of reaction time) after the channel cross. The second location was positioned 26 mm (equivalent to ca. 20 s of reaction time) after the channel cross. A temporal evolution of the structure in the form of a swelling of the lamellar phase was observed as shown in Figure 2. The X-ray data analysis with Scatter yields distances of the lamellae of 9.2 nm in the front and 11.9 nm in the back of the channel.

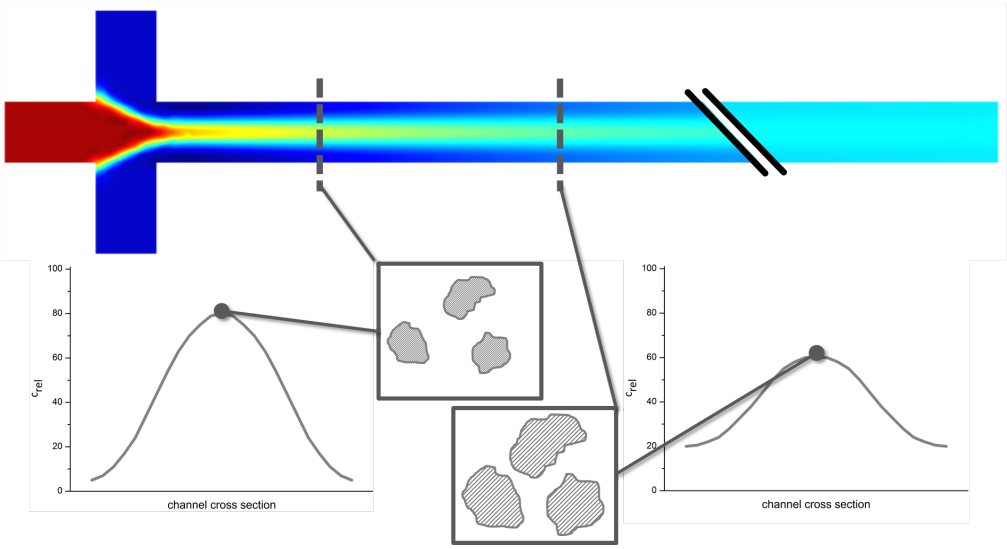

Figure 2: concentration profile in the microfluidic channel and a scheme of the temporal evolution of lamellar structures

The microfluidic platform we presented allows for kinetic studies on nanoscale materials. In the future, the possibility to investigate kinetic effects down to microsecond scale depending on flow-velocity via SAXS may help to understand nucleation und growth processes in nanoparticle synthesis. This makes it possible to improve the concept of synthesis by changing from empirical optimization to a rational and predective strategy.

References

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