A thin liquid-jet and the microstructure of elongated particles

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The ability of modern synchrotron and XFEL light sources to produce highly intense coherent X-ray beams in the µm and sub-µm range offers the possibility to image soft condensed matter on ultrasmall length and ultrafast time scales. This ability allows to access a variety of coherent X-ray diffraction concepts e.g. X-ray Photon Correlation Spectroscopy (XPCS) and X-ray Cross Correlation Analysis (XCCA) [1, 2, 3].

The highly intense beam and the vacuum conditions in such experiments imply constraints on the sample housing, e.g. radiation damage of the samples or the walls of the sample container may affect the sample structure. One possibility to overcome this problem is a steady streaming injection system that produces a thin liquid-jet in vacuum.

Liquid-jets offer the possibility to study disordered samples in the fluid phase by a very fast steady streaming [4]. We designed a liquid-jet setup which is capable of producing a homogeneous thin liquid-jet of thicknesses down to 1 µm. In this experiment we studied the small angle X-ray scattering (SAXS) structure of elongated nano-particles as a prototypical application of our liquid-jet setup. An overview about the setup is depicted in figure 1. We used a microscope and a mirror at the optical axis of the x-ray beam to align the thin liquid jet to the x-ray beam. This mirror provides a small hole for the x-ray beam. We used the Pilatus 300k detector at a sample detector distance of 5 m at beam line P10. Due to the small thickness of the jet we performed the measurements with a focused x-ray beam of 3 µm x 6 µm at an energy of 7 keV.

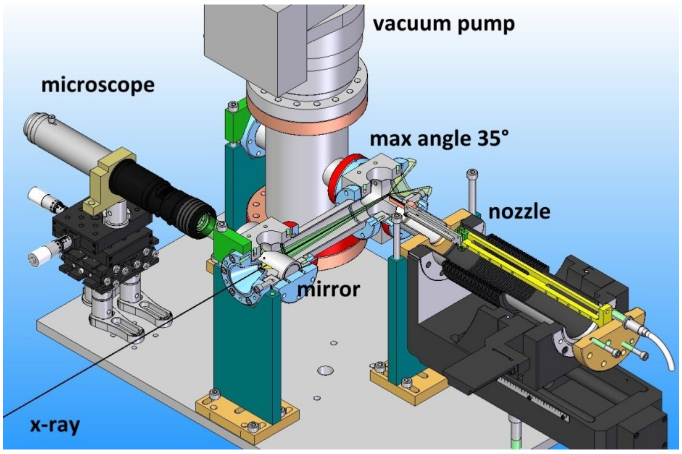

Figure 1: Sketch of the used setup. The x-ray beam illuminates the jet from the left lower corner.

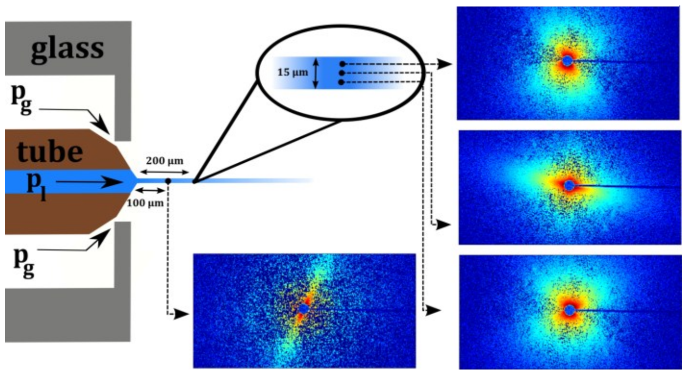

Figure 2: Schematic overview of the measured SAXS patterns at different jet locations relative to the nozzle tip.

In this way we were able to take spatially resolved SAXS patterns at different locations of the jet. The experiment was performed with a highly diluted colloidal solution (0.1 vol.%) of elongated particles. Thus the measurements are dominated by the form factor and thus by the orientation of the particles in the jet. An overview of the different measurements is shown in figure 2. A strong particle alignment 100 µm below the nozzle tip produced a narrow SAXS signal. While at 200 µm distance this strong alignment is disturbed. At the jet sides X-shape like patterns are visible which point to a two-state orientation of the particles at this location. In the middle of the jet an orientation perpendicular to the flow is visible. This can be understand in terms of shear alignments of the particles. These results are in line with other rheology measurements on elongated particles [5].

Moreover we measured a density modulation in a few cross-sections of the jet close to the nozzle tip, see fig. 3. The summed scattering intensity which is a measure of the electron density provides two maxima at the jet sides. This can be a hint for a clustering effect of the particles at these positions. The extracted jet thicknesses as function of the nozzle distance shows a changed slope at 500 µm nozzle distance which can be connected to a possible break-up of the jet (Fig. 3, right).

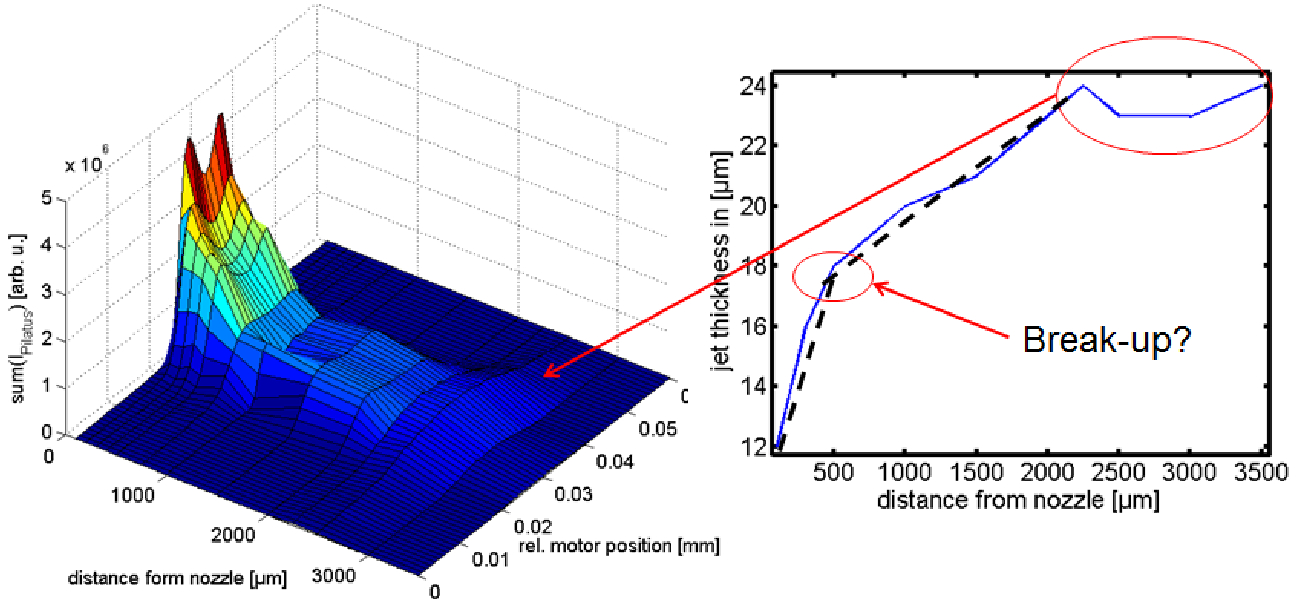

Figure 3: Left: Sum of scattered photons on the Pilatus detector as function of the position on the jet and distance from the nozzle tip. Right: Jet thickness as function of the nozzle distance. Due to low statistics the determination of the thickness above 2000 µm nozzle distance is difficult.

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