ESRF	**Experiment title:** *In situ* investigations of adhesive spider hairs using nanodiffraction	**Experiment number**: SC-3968
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Report:

Using scanning X-ray nanodiffraction we investigated the attachment process of adhesive hairs of the hunting spider *Cupiennius salei in situ*. These hairs located on the feet enable the spider to climb smooth vertical surfaces as well as to walk upside down on the ceiling. This ability bases on bringing the most distal substructures at the tips of the hairs in close contact with the substrate leading to adhesive van der Waals forces.
In continuation of the static structural studies of beamtime SC-3736, single hairs mounted on the tip of a micropipette were brought in contact with a fixed silicon nitride window positioned in the X-ray beam by means of a nanometer precise manipulator (Fig. 1a). Scanning of the samples in both the unattached and the attached state was performed in combined wide angle X-ray scattering (WAXS)/small angle X-ray scattering (SAXS) mode. Mapping the diffraction patterns was aimed for the reconstruction of changes in fiber orientation in the substructures located at the tips of the chitin-containing adhesive hairs.

The X-ray diffraction experiments were performed at the nanofocus endstation of the beamline ID13 using a beamsize of 250 × 250 nm and beam energy of 14.85 keV. Overall, four adhesive hairs of the spider Cupiennius salei were scanned both free-standing (unattached) and attached and to the surface of the Si_3N_4 window serving as the substrate. The experiments resulted in nine scans of four samples. One sample was also slightly sheared after measuring the attached state and scanned again. The scan comprised areas of 20 × 20 µm to 30 × 30 µm, and resulted in both SAXS- and WAXS signal maps consisting of up to 14641 diffractograms. The scans were performed with the direction of the beam in line with the direction of attachment except one, where the tip of the attachment hair was scanned from the side (Fig. 1b).

The analysis of the WAXS-signals showed a diffuse circular signal, which indicates the amount of the sample in the beam. A shape change of the hair from the free-standing and the attached hair is clearly visible. In contact with the substrate, the terminal contact elements of the hairs are attracted to the surface of the Si_3N_4-window resulting in a smaller visible thickness of the hair (Fig. 1c).

The SAXS-signals also contain information about the orientation of the hairs´ substructures. The analysis of the angle of orientation shows at least partly an orientation change of the contact elements of the hair by up to 90 degrees (Fig. 1c). Further analysis focusing on the Herman´s orientation parameter is underway.

The intensity of the scattering signals was astonishingly intense especially considering the small volume of the samples. The high resolution of the scans allow for proper reconstruction of the fine details of the functional hair tip. Handling the minute spider hairs *in situ* was comfortable after setting up of the manipulator remotely controlled from the control hutch. The planned continuation of the experiment will focus on the side aspect of the hairs, where the change of orientation of the single contact elements may be visible even more clear, and a miniature force sensor will be used for force controlled attachment and detachment.

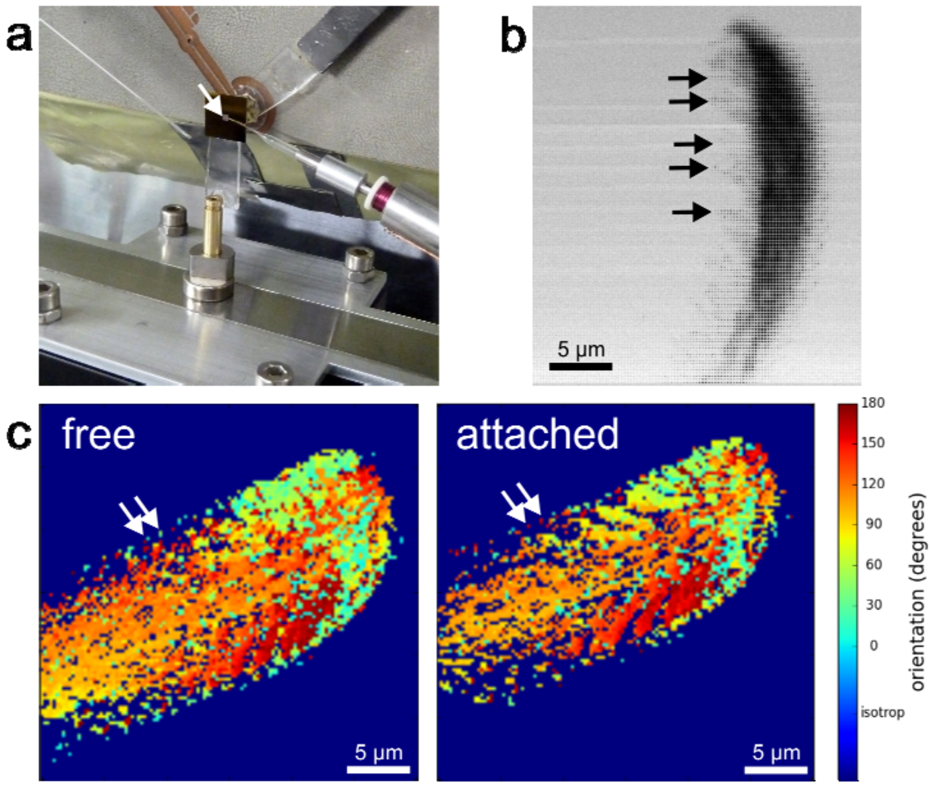

Figure 1 (a) Setup of the spider hair (arrow) mounted on the tip of the manipulator for *in situ* experiments at ID13. (b) Map of the SAXS-signals of the functional tip of a spider attachment hair seen from the side. The arrows point to single contact elements. (c) Orientation of the SAXS signal of a hair before (left) and in contact (right) with the Si_3N_4 substrate. The arrows point exemplarily to single contact elements which change their orientation by about 90 degrees due to attachment of the hair.