ESRF	**Experiment title:** Evolution of the silk microstructure in the Aranae lineage (spiders) and comparison with bioinspired fibers”	**Experiment number**: SC3325
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Shifts: 9	**Local contact(s)**: C. Riekel, M. Burghammer	*Received at ESRF:*

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Report:

Four sets of materials were studied: (1) spider major ampullate gland silk (MAS) from different Aranae lineages, (2) Flagelliform silk from the spider *Argiope trifasciata*, (3) Silkworm (*Bombyx mori*) silk fibers subjected to different physicochemical treatments, and (4) Regenerated silkworm silk fibers produced by wet spinning.

(1) MAS silk from different Aranae lineages.

Silks are one of the defining traits of spiders (Aranae), with each spider species producing at least one type of silk. Present silks have evolved from a common ancestral gland for almost 400 million of years but, despite the obvious interest, only now comparative studies can be undertaken to establish the similarities and differences between different silks. MAS silks from six different species that represent the major spiders groups were analyzed and their basic microstructural features determined.

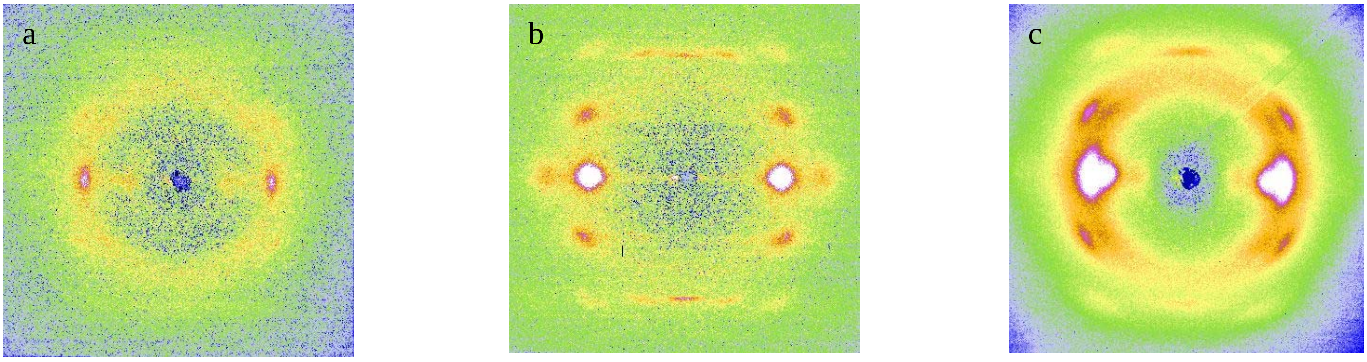

Figure 1. XRD diffraction pattern of maximum supercontrated MAS fibers from (a) *Aphonopelma seemani* (Mygalomorph), (b) *Kukulcania* sp. (Haplogyna), and (c) *Caerostris darwinii* (Entelegyna).

(2) Flagelliform silk of the spider *Argiope trifasciata*.

Flagelliform (Flag) silk is used by orb-weaving spiders to build the spiral of the web. Flag shares some basic motifs of sequence with MAS, but lacks the polyalanine runs that make up MAS nanocrystals. Consequently, there was an argument on the possible semicrystalline nature of Flag, as opposed to models that favoured a completely amorphous microstructure. Besides, it was found that Flag undergoes supercontraction and, in parallel with MAS, it is possible to control their properties predictably and reproducibly by stretching Flag supercontracted fibers. Stretching is measured with the alignment parameter, defined as the ratio between the final length of the fiber after stretching and the initial length of the supercontracted fiber. XRD diffraction patterns of Flag fibers with different values of the alignment parameter were obtained showing not only the semicrystalline character of Flag, but also the evolution of the crystalline phase with stretching.

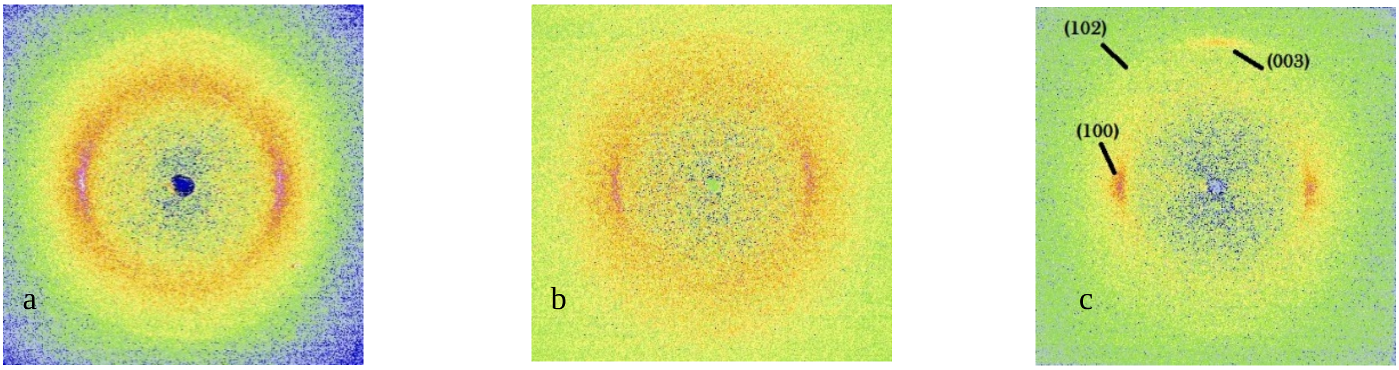

Figure 2. XRD diffraction patterns of Flag fibers with different values of the alignment parameter, α. (a) α=0, (b) α=0.45 and (c) α=0.9.

(3) Silkworm (*Bombyx mori*) silk fibers subjected to different physicochemical treatments.

The evolution of the crystalline phase of *B. mori* silk subjected to different physicochemical treatments was analyzed. Silk retrieved by forced silking was used as control samples. The treatments included immersion in water and degumming, i.e. immersion in boiling water. Significant changes in the diffraction patterns were observed.

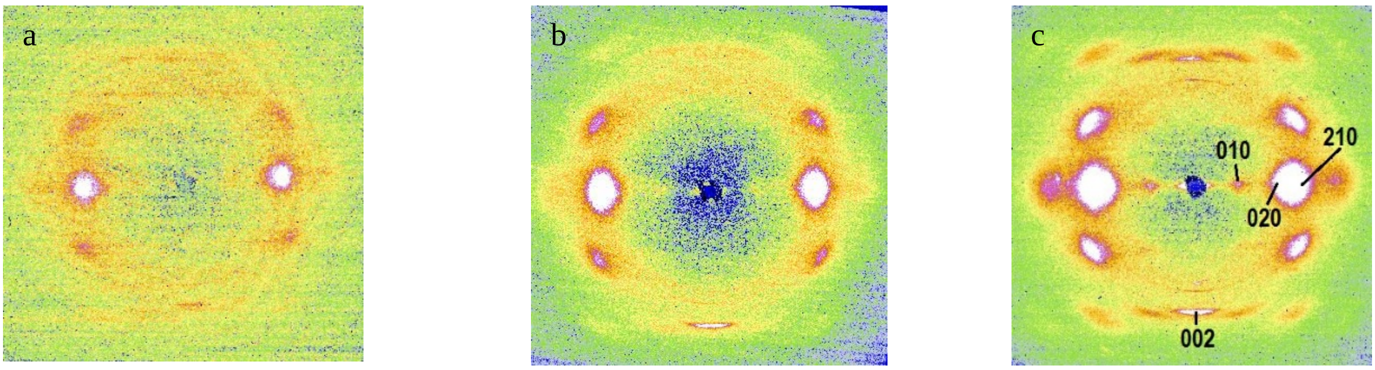

Figure 3. XRD diffraction patterns of *Bombyx mori* silk fibers (a) obtained by forced silking (control sample), (b) immersed in water for 30 minutes, and (c) degummed in water for 30 minutes.

(4) Regenerated silkworm silk fibers produced by wet spinning. XRD analysis revealed the absence of a crystalline phase suggesting an amorphous microstructure for these fibers.