ESRF

Experiment title:

Structural Studies on Single Spider Silk Fibres by X-Ray Microdiffraction

Experiment number:

LS369

Beamline:

ID13

Date of experiment:

from: 4.6.96 to: 6.6.96

Date of report:

29.8.96

Received at ESRF

Shifts:

9

Local contact(s):

A. Brain

Names and affiliations of applicants (* indicates experimentalists):

C.L. Craig* Yale University, Department of Biology

C. Riekel* ESRF

C.I. Bränden* ESRF

REPORT

A range of different varieties of spider silk -mostly collected from freely foraging spiders- was available for the experiments. In the following we will concentrate mostly on results obtained from *Kukulcania hibernalis -an* ancestral spider- and *Nephila clavipes* - an orb-spinning spider. In both cases single fibre diffraction patterns could be obtained for the frost time.

All spider silk was characterized at ESRF optically and by SEM methods. For diffraction experiment samples were fixed on electron microscopy apertures. Wide-angle X-ray scattering (WAXS) experiments were performed using the single crystal diffractometer of the *microfocus beamline. The beam* size was 30 microns and the wavelength 0.1488 nm. All experiments were performed at r.t. which resulted in a degradation of the scattering pattern -as observed on-line by an image intensified CCD-camera- in ≈ 30 sec. Fig. 1 shows the diffraction pattern of a *Kukulia hibernalis* single fibre of 4 μm diameter recorded in 12 seconds. Reflections observed extend to the second layer line. Several equatorial and first layer line reflections could be qualitatively fitted by 2D-Gaussian profiles. Reflections can be indexed according to the unit cell of *Bombyx mori(Marsh* et al., 1955; Warwicker, 1960; Takahashi, 1994). The same is the case for the pattern of a 5 μm *Nephila clavipes* fibre. It is interesting to note that the lack of a 200-reflection in N. *clavipes* would put this species into a new group according to the classification of Warwicker(1960). These experiments are very encouraging as it has been demonstrated that the flux on the microfocus beamline is sufficient to obtain significant diffraction patterns on weakly scattering fibres in a few seconds.

In order to obtain higher resolution' data it will be necessary to stabilize the fibres. There are 2 options for this: (i)by translating the fibre during experiments or (ii)by Cryocrystallography techniques. The latter technique is preferable as there are indications for a variation in scattering power along the fibre axis for some of the samples studied.

The data on these two fibre-samples do not show a strong diffuse scattering background. Such a finding would have been of interest in the context of the proposed semicrystalline morphology of spider silk which has been proposed to explain its exceptional mechanical properties (e.g. Termonia, 1994). A strong diffuse halo was ,however ,observed in the WAXS pattern of a well aligned fibre bundle from *Argiope argentata* (Fig.2). In this case it would be of interest to use small-angle scattering techniques -preferentially from a single fibre- for further morphological studies.

References :

Marsh, R.E. et al., 1955, Acts Cryst., 8,7710

Warwicker, J.O.,1960, J. Mol. Biol.2,350-362

Takahashi,Y. in *Silk Polymers,* Amer. Chem. Sot., Washington DC, 1993

Termonia, Y.,1994, Macromolecules, 27,7378-7381

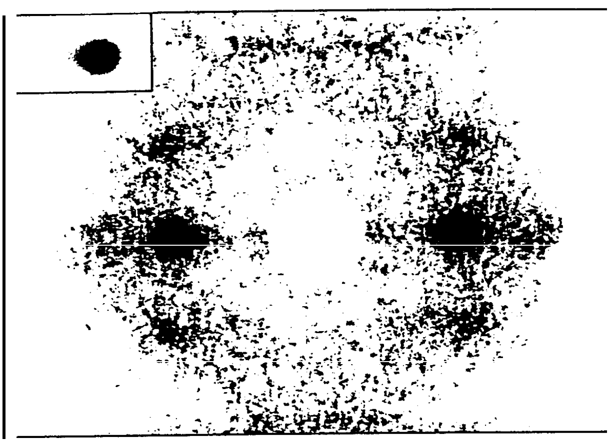

Fig.1 *Kukulia hibernalis* single fibre -12 sec fibre axis vertically inset shows fit to equatorial (120)/(210)

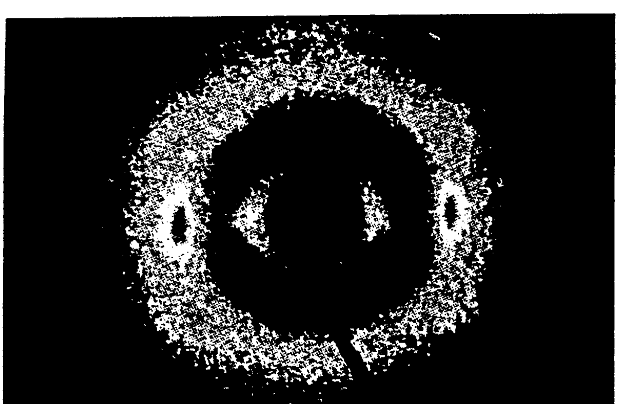

Fig.2 *Argiope argentata* fibre bundle - 12 sec fibre axes vertically