ESRF	Experiment title: Biomimetic Nanotubes : Structure and functionalisation Studied by SAXS/WAXS Fiber diffraction	**Experiment number**: SC 1246
Beamline: ID2	**Date of experiment**: from: 30 january 2004 to: 2 february 2004	**Date of report**: 17 march 2004
Shifts: 9	**Local contact(s)**: Thomas WEISS	*Received at ESRF:*

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Report: The wavelength energy was 12.0keV with a the beam a size of 200x200μm^2 on sample and on detector. Attenuation was only due to the beam definition slits. Two SAXS detectors were used, the CCD and the Image Plate. In the case of the CCD detector, the sample/detector distances were 1.2m, 3m, 6m. In this condition, the accessible scale was q=0.005-0.5Å^{-1}. The WAXS detector was simultaneously used with a q-range of 0.9-3Å^{-1}. The 9 shifts, 72 hours, were used as described in the following :

A] beamline alignment, setup installation and calibrations .

B] **sample stability tests**. Under these conditions, silica nanotubes are stable with pulse longer than 200ms. Radiation damages are observed only after a few seconds. In this conditions the acquisition time is limited by the detector saturation.

C] **Structure of Biomimetic Nanotubes**. A wide variety of samples were investigated in order to check some hypothesis : i) It is possible to tune the diameter of the Lanreotide nantotubes [1], by the mixture of octapeptides with analoguous, by adding salt in water. ii) X-ray diffuse scattering of the first step of association of the beta-sheet filament into nantotubes are in agreement with first EM experiment (helicoidal ribbons).

C] **functionalisation of Biomimetic Nanotubes**. The condition of functionalisation of the nanotubes by silica precursors has been studied systematically by SAXS (more than 100 samples). This let us to find reproductible preparation conditions of well oriented silica nanotubes. Some of the sample were consecutively studied by Electron Microscopy (Fig. 1a).

Figure 1b and 1c demonstrates i) the perfect parallelism of the fibers (mosaicity < 5°) ii) the monodispersity of the diameter (14 oscillations are observable) iii) the thickness monodispersity of the deposited silica 15Å (good fit agreement). Surprisingly, the silica is deposited on both inner and outer face of the nanotubes.

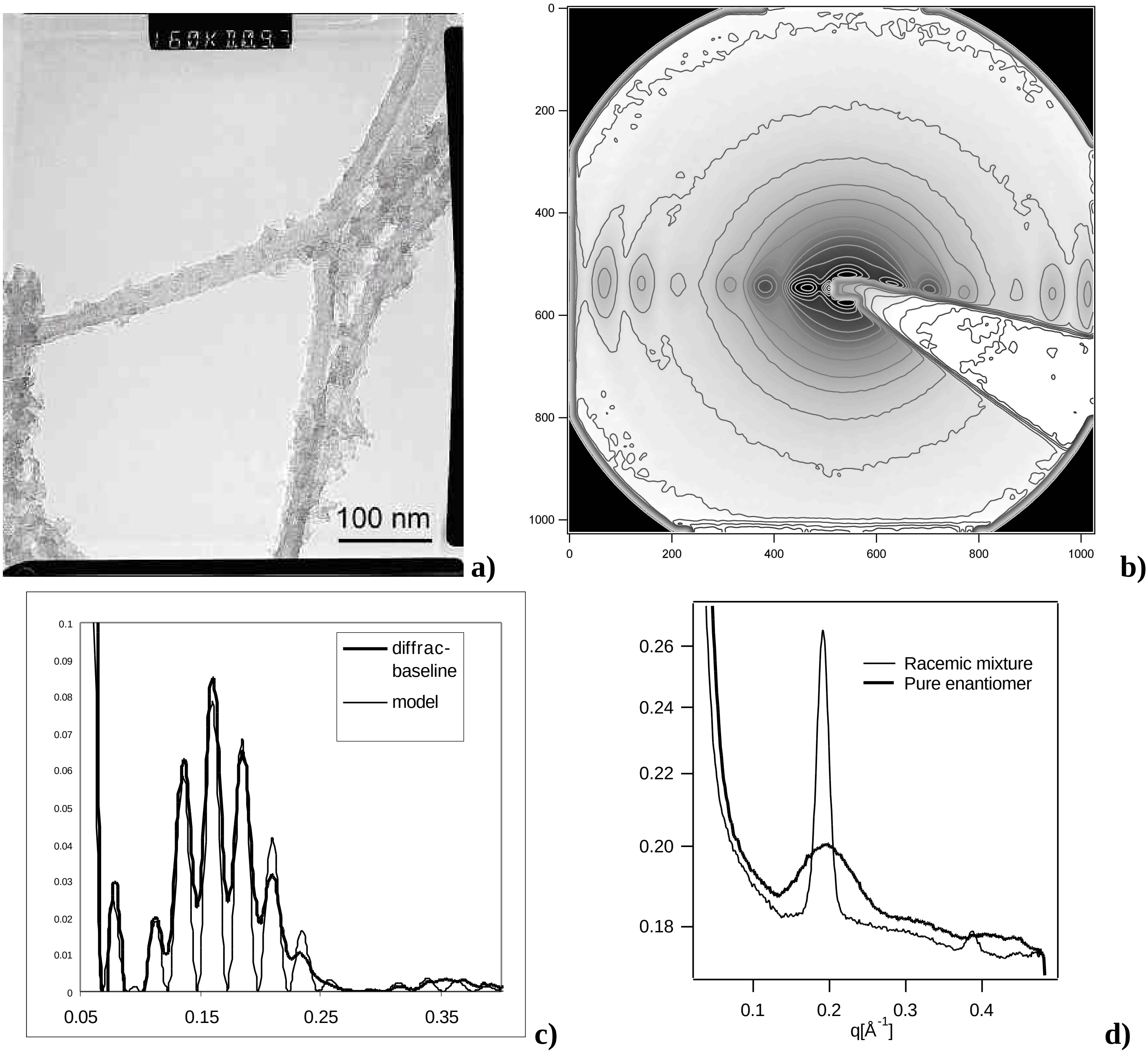

Figure : a) Electron microscopy of silica nanotubes (E. Dujardin at CEMES toulouse). b) X-ray fiber diffraction of well-oriented silica nanotubes (d=3m). c) Powder X-ray scattering (1.2m) and theoretical fit of silica nanotubes with a silica thickness of 14 Å d) chirality effect on the supramolecular organization of tartrate-gemini (data collected at 1%).

E] Chiral fibers : Two chiral systems have been tested in collaboration with J. Malthete (Insitut Curie, Paris) and Reiko Oda (IECB, bordeaux) [2]. Both preliminary experiments have been succesfull and evidence effects of the chirality on the supramolecular organisation (see for example Fig. 1d). The next step will be to correlate these molecular effects with the morphologies.

1] Valéry, M. Paternostre, B.Robert, T. Gulik-Krzywicki, T. Narayanan, J.-C. Dedieu, G. Keller, M.-L. Torres, R. Cherif-Cheikh, P. Calvo & F. Artzner, *Biomimetic organization : octapeptide self assembly into nanotubes of viral capsid like dimension*, **Proc. Natl. Acad. Sci. USA,** 2003, 100(18), 10258-10262
2] R. Oda, I. Huc, M. Schmutz, S. J. Candau, F. C. MacKintosh, *Tuning Bilayer Twist Using Chiral Counter-Ions*, **Nature** 1999, 399, 566.