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| | Experiment title: Evaluation of breast cancer and auto-immune diseases diagnosis from hair microstructure | Experiment number: LS-1570 |
| Beamline: ID13 and ID21 | Date of experiment: scheduled in April and May 2000 Tests carried out on September and November 1999 | Date of report: February 2000 |
| Shifts: | Local contact(s): C. Riekkel and J. Susini | <i>Received at ESRF:</i> |
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REPORT

On the last round (August 1999), we submitted a long term proposal (2 years) entitled "Evaluation of breast cancer and auto-immune diseases diagnosis from hair microstructure". We were rewarded 15 shifts on ID13 and 15 shifts on ID21 for experiments to be carried out during the first semester of 2000, and were asked for an interim report for continuation for the next rounds. *Since our experiments are scheduled on next April and May, we are not able to provide today a report about this first set of measurements. However, it is of prime importance for our project to get beamtime evenly in order to be able to analyse the data as soon as possible and to direct the next hair collections according to the first results.* Let us recall that the first year of our project was dedicated to microdiffraction and micro-chemical analyses in order to evaluate the most significant parameters, the most efficient techniques and the most promising applications for diagnosis.

However, we got a few hours beamtime to carry out tests experiments on ID13 and ID21 on september and november 1999. These tests were too short to evaluate the most significant parameters or the most promising applications for diagnosis, but they were long enough to evaluate the technical feasibility of our project and to precise the best experimental conditions. The results are quite positive, they clearly prove the feasibility of our project on ID13 and ID21.

Microdiffraction experiments

Experiments were carried out on the microfocus beamline ID13 with a 13 keV energy beam size-limited down to a 10 μm diameter section by a collimator. A pinhole was added close to the sample to remove scattering signal from air. Samples were mounted on a computer-controlled Physik Instrument X/Y stage coupled to a microscope which permitted to position them in the beam with 0.1 μm spatial resolution. The step size between data points was 10 μm . Patterns were recorded on CCD MARRESEARCH camera located at 240 mm from the sample using 10 s exposure time. Using a small diameter beamstop (0.3 mm) located about 40 mm from the sample allowed us to collect both SAXS and WAXS data in the 150Å–2Å region. Therefore, the main scattering features are visible on a single pattern of an hair perpendicular to the

beam. They consist in a broad equatorial spot centered at 9.7 \AA , corresponding to the mean distance between α -helical axes, a fine meridian arc at 5.15 \AA , related to the projection of the α -helix pitch along the coiled coil axis and broad equatorial peaks at 91 \AA , 45 \AA and 29.5 \AA due to the dense lateral packing of microfibrils. On the meridian, the most intense reflection is seen at 67 \AA , it arises from the axial stagger between molecules along the microfibril. The ring arising from lipid granules is located at 45 \AA .

Six samples were analysed, two typical scattering patterns are shown in fig. 1. All scattering features are visible in fig. 1-a (SAXS region of healthy person) whilst they are very weak or missing in fig. 1-b (SAXS region of sick person). Huge differences can thus be sometimes observed between the patterns from healthy and sick person; a statistical study is necessary to establish correlations and reach medical conclusions.

We have designed a sample holder containing about 50 hairs that are located at regular 1 mm intervals. The automatic data collection using such a sample holder should require about 2 to 3 hours assuming a transverse mapping with a $10 \text{ }\mu\text{m}$ step.

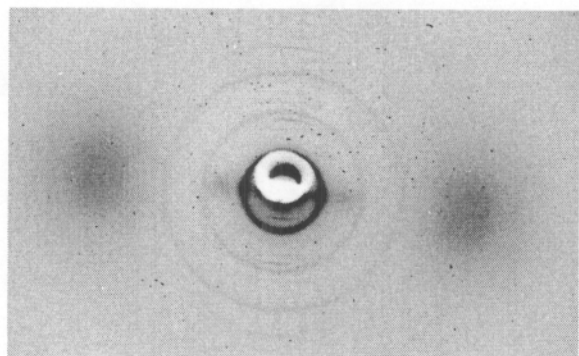


Figure 1-a



Figure 1-b

Micro-XANES mapping experiments

Micro-XANES mapping tests on human hair sections were carried out on ID21 around the sulphur edge (2.45 keV) on the Scanning X-ray Microscope. The beam was size-limited by a zone plate focussing lens and a pinhole aperture (selecting the third order) down to $0.25 \times 0.25 \mu\text{m}^2$. The hair sections were raster scanned with a pixel size of $1 \mu\text{m}$ at an energy corresponding to a peak of the XANES spectrum with an energy resolution of $\Delta E/E = 10^{-4}$. Absorption images were also obtained to correct from possible thickness irregularities. Data collections on pure cystin and cystein compounds were performed to get standard spectra of the two major chemical states of S atoms in proteins. The three zones of human hair, i.e. the cuticle, the cortex and the medulla, can be seen on the images (Fig 2). The technique can therefore be applied for looking at possible S content changes according to natural variability or pathologies.

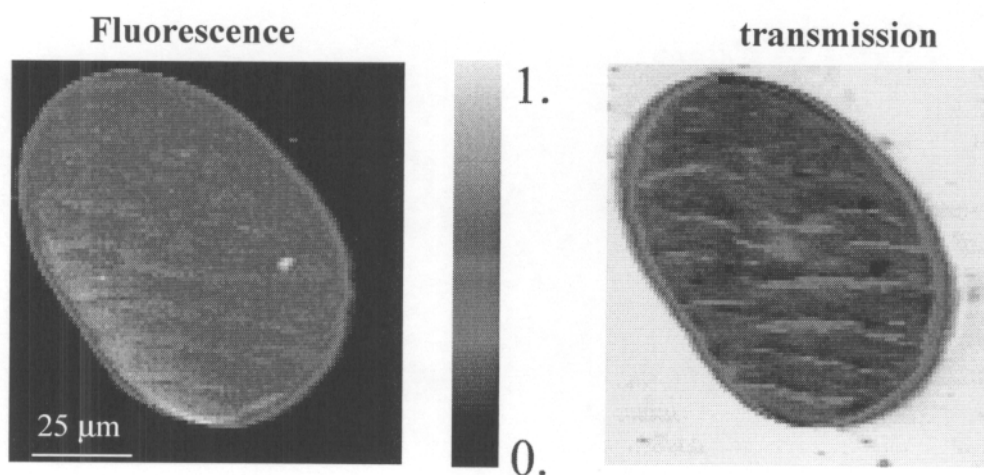


Figure 2