ESRF	**Experiment title:** microXANES and microXRF investigation of the Fe-Si relationships in human lung tissue affected by silicosis	**Experiment number**: CH 4920
Beamline: ID21	**Date of experiment**: from: October 26th, 2016 to: October, 31th, 2016	**Date of report**: 10/09/2017
Shifts: 18	**Local contact(s)**: Giulia Veronesi	*Received at ESRF:*

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Report:

Introduction

Object of the CH4920 experiment was the study, through a combined μXRF and μXANES investigation, of the distribution of Fe and Si particles in an autoptic human lung tissue that belonged to a worker affected by silicosis. During this characterization, the eventual co-localization of the two ions, and eventual changes of Fe speciation and its distribution in the tissue were also investigated.

Materials and experimental procedures

The tissue sample was prepared as a series of 10 μm thick histological sections supported on ultralene polymer, and as conventional 30 μm thick colored histological sections supported on glass and polycarbonate. The former sections were used to the X-ray characterization at the beamline, while the latter were used to a companion screening carried out at the visible microscope, also available at the ID21 beamline.

After having analysed (in both the XANES and EXAFS regions) a series of relevant standards (including ferritin and hematite), we performed several X-ray Fluorescence maps according to the following parameters: Energy of the X-ray radiation: 7.28 KeV; panoramic maps (5 μm x 5 μm), zoom maps (1 μm x 1 μm), detail maps (0.5 μm x 0.5 μm). The size of each map (i.e. the size of the scanned region) was balanced to achieve a total counting time not higher than 4 hours. Then, XANES spectra were registered in numerous regions of all the registered maps.

At the end of the experiment, i.e. during the last two shifts, two attempts of registering an hyperspectral μXRF-μXANES map were performed, on selected detail maps, previously noticed as interesting for the Fe-Si co-localisation and for the Fe speciation. The conditions for the registration of the hyperspectral maps were the following: detail map (0.5 μm x 0.5 μm), energy range 7-7.28 KeV. The maps, each one registered at a fixed monochromatic energy value, were then stacked by means of a software developed by the beamline staff.

Preliminary results

The preliminary results obtained are summarised in the Figures here below.

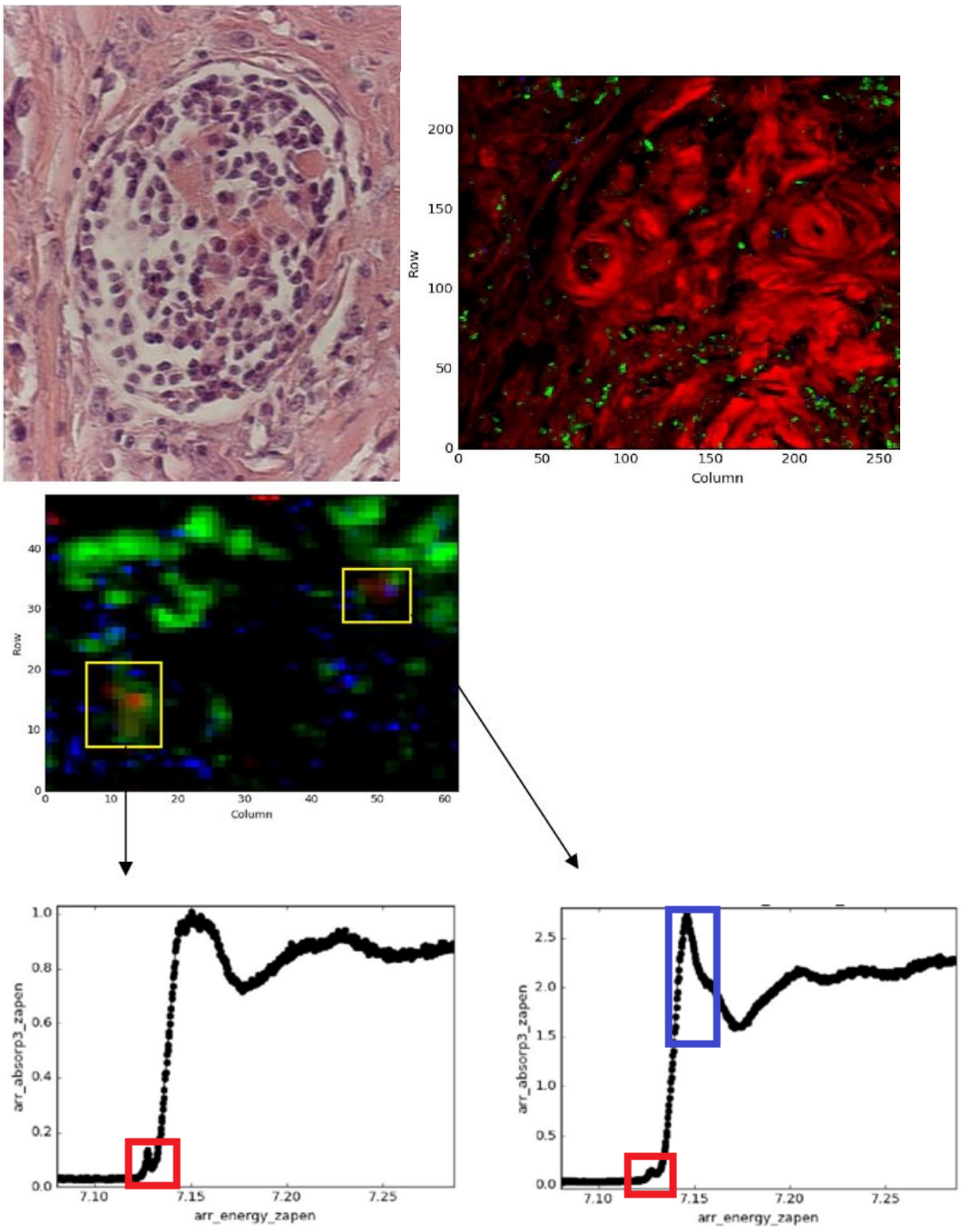

Figure 1 – Top left – example of a silicotic nodule (visible microscopy); top right – example of a μXRF map (red – S distribution, green – Si distribution); middle – detail map (red – Si distribution, green – Fe distribution; blue – Ti distribution); down left and right – exemplar XAS spectra obtained in the different regions marked by the yellow rectangles.

- The feasibility of the experiment has been verified: silicotic nodules were identified by (transmittion) optical microscopy, and re-identified by means of the combined reflection optical microscopy

(present in the experimental hutch) and μXRF on a different thin section mounted on ultralene (Figure top left and top right)
- Acquisition of detailed high magnification maps (Figure middle)
- Identification of at least two different species of Fe(III), one of which, (Figure down left) traces an apparent tetrahedral coordination; the other one, is consistent with the ferritin standard (Figure down right)

From the above consideration, interpretation is in progress to verify if the tetrahedral Fe(III) can be attributed to an inorganic species, and to verify its colocalisation with Si.