EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

# Experiment Report Form

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| | **Experiment title:**<br>"Study of metallic element released by joint implant and determination of the oxidation degrees " | **Experiment number**:<br>LS-2129 |
| **Beamline**:<br>ID-21 | **Date of experiment**:<br>from: 09/02/02 to: 14/02/02 | **Date of report**: |
| **Shifts:**<br>15 | **Local contact(s)**:<br>Murielle SALOME | *Received at ESRF:* |

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**Report:**

We worked on capsular per-operatory knee sample and post-mortem hip sample. Preliminary analysis by spark spectrometer give the composition of the prostheses and permit to know which kind of element we can encounter in the tissues. Knee prosthesis is composed of two types of implant. The femoral part is in titanium alloy (Ti-V) and the tibial part in cobalt alloy (Co-Cr-Ni-Mn). The hip prosthesis is in stainless steel. The samples are conserved in liquid nitrogen. Tangential cut are realised with a cryomicrotom and deposited on PEEK support recovered by a film of formvar.

The experimental settings up to optimise the analysis were very difficult. We had problem with the stability of the beam bound with the thermal instability of the monochromator, which induced a gap of the microbeam between the different scans on the sample. We are interested by chromium whose toxicity and carcinogenic effects are known. But the tissues which were in contact with the two types of alloy (titanium and cobalt alloy) presented a high concentration in titanium which saturated the germanium detector and interfered with the chromium cartography. These samples cannot be studied by micro-XANES in point of fact of the high concentrations.

The first acquisition was realised with a zone plate of 1040 µm of diameter which give an important flux and a better sensibility to chromium. The beam diameter was around 3 µm. The obtained cartographies show the

possibility of co-location of different elements, mainly for the chromium and the titanium. The micro-XANES was also very difficult because of the instability of the microbeam.

We were then interested to the samples taken near stainless steel implant. The higher concentration in chromium induced the saturation of the detector and a lot of dead time during the acquisition. We used aluminium filter in order to reduce the incident beam, but we have the problem of beam stability. We realised cartographies which give informations about the surface repartition.

The sample presented a chlorine signal, which can be easily attenuated by a kapton film, and a chromium signal. We replace temporally the dispersive detector of fluorescence by a photodiode for the acquisition of fluorescence spectra in concentrated zone. With this method, we have not the problem of saturation. By observation of the spectra, we notice that the threshold of the chromium is different between two positions.

In order to improve the stability of the microbeam during the acquisition of the cartographies and the micro-XANES, we change the zone plate and take the one of 69-µm diameter which have a better resolution (< 1µm). The small diameter of this lens allows the shutting of the split which reduces the thermal charge on the monochromator. Furthermore, the focal, 10 less than the other, limits the sensibility of the position of the microbeam opposite to the instability of the monochromator. The cartographies (figure a) show a better resolution and a better definition of the different grains encountered. The micro-XANES spectra are more reproducible than those obtained before (figures b and c). We think that we can determine the nature of chromium with this method.

During these experiments, we analyse different references with chromium. We have used $K_2Cr_2O_7$, $Cr_2O_3$ and $CrKO_8S_2$, and a solution of $Cr(NO_3)_3$. In addition, we have made micro-XANES on sample of prostheses.

This first experiment shows the feasibility and the adequation of this method to do the microspectrometry of chromium in tissues. Complementary experiments are necessary to verify the generality of these data on a great number of samples.

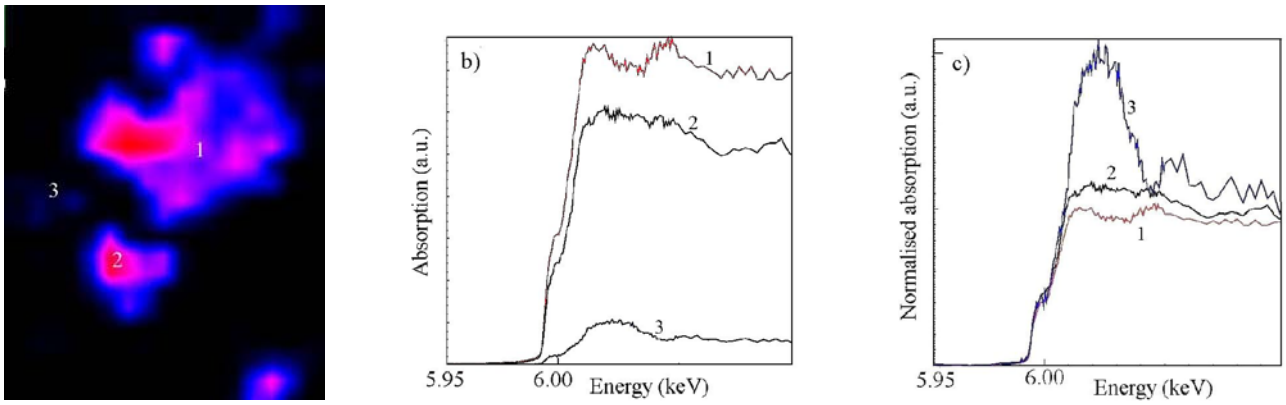


Figure a: Cartography of a grain at different points: 1, 2 and 3.
Figure b: Absorption spectra obtained for the different points (red =1, black = 2, blue = 3).
Figure c: Normalized absorption spectra (red =1, black = 2, blue = 3).