	Experiment title: X-ray microdiffraction with high spatial resolution to sudy interface of zirconia prostheses with bone	**Experiment number**: MI490
Beamline: ID13	**Date of experiment**: from: 01 July 2002 to: 07 July 2002	**Date of report**:
Shifts: 12	**Local contact(s)**: Manfred Burghammer	*Received at ESRF:*

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Report:

In the course of the long term proposal MI 490 we have carried out until now two sessions on ID13, one from 28 June 2001 to 02 July 2001 and the second from 01 July 2002 to 07 July 2002.

These two sessions have been mainly used for microdiffraction studies to analyse the interface between a Zr prosthetic device implanted in a rat femur and the newly-formed bone, with a spatial resolution of 0.5 micron. The experiments exploit the innovative X-ray microdiffraction technique based on the X-ray waveguide developed by our group.

In particular while the first session had mainly methodological objectives, the second session has been used to make a complete study of new-bone quality related to the particular kind of implanted prostheses. In particular the osteointegration role of the coating process has to be structurally studied. For this porpoise Zr samples with RKKP bioglaze® coating have been compared with samples without coating. Figure 1 shows the intensity variation of the reflections of OCP and HA at the interface region for coated (a) and uncoated (b) samples.

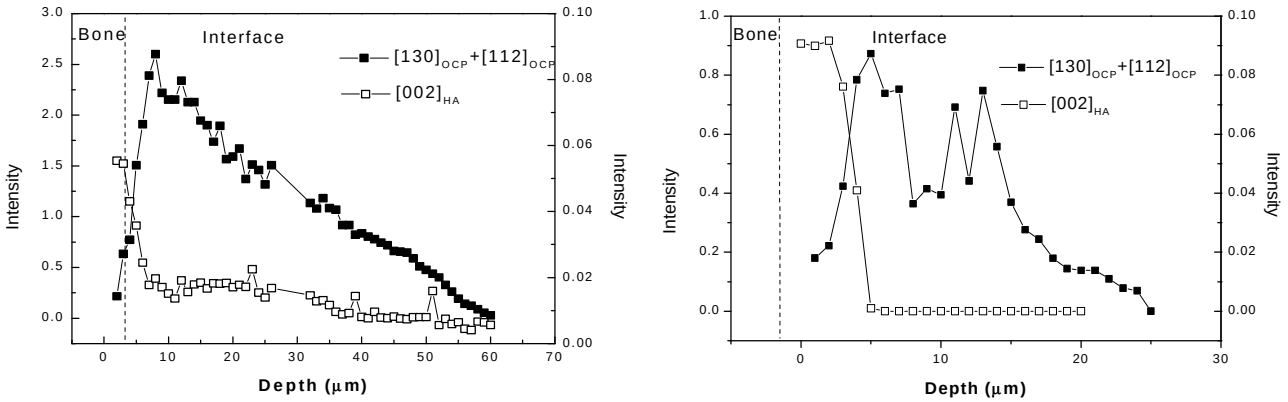


Figure 1. Intensity variation of the reflections of OCP and HA at the interface region for coated (a) and uncoated (b) samples.

As it is clear from the figure, in the coated sample the interface region hosts both the OCP and the HA crystallographic phases, while in the uncoated sample only the OCP is present, indicating that the transformation of OCP in HA is accelerated by the RKKP (osteointegration process). Further analysis is still in progress.

In this second section we also made a micro-diffraction test experiment dealing with problems related to cultural heritage conservation. The sample was a travertine section coming from the Marcello's theatre in Rome. The aim of the experiment was the study of the degradation process induced by the aggressive atmosphere of SO_2 on the carbonate rocks ($CaCO_3$). The experiment intended to measure the concentration profile of $CaSO_4$ produced in a layer below the external surface due to the presence of SO_2. Figure 2 shows the geometry of the experiment: A thin section of the rock is scanned, from the surface to the inner region, through the micro-beam and microdiffraction pattern is recorded at each position.

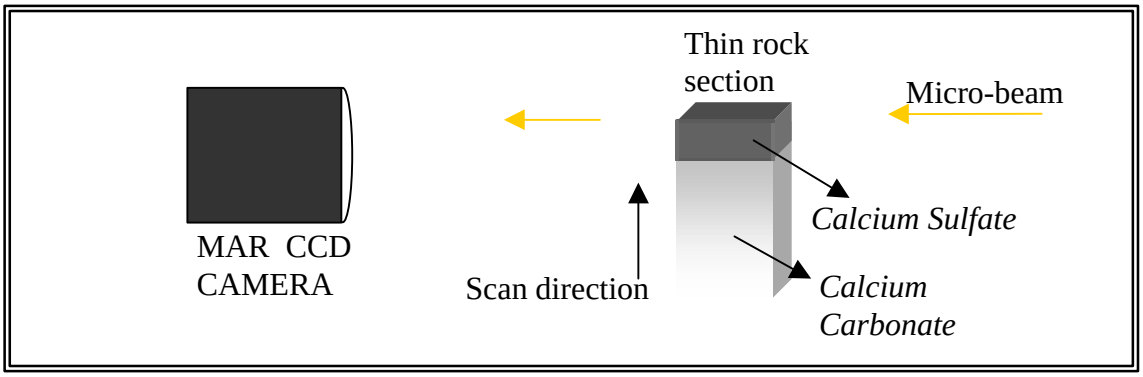
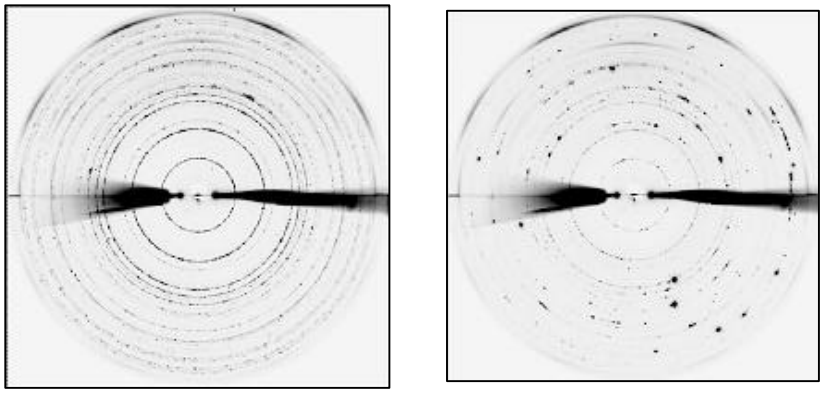


Figure 2. Geometry of the experiment

Diffraction patterns recorded from two different depth positions, along the scan direction (Fig.3a and b) allow one to determine the difference in structure (crystal lattice spaces) and morphology (crystallite dimensions) of the two diffracting regions, indicating the possibility to follow the $CaSO_4$ concentration profiles. In this case an interface region few hundred microns thick where the $CaSO_4$ and $CaCO_3$ phases coexist has been determined.



a) b)

Figure 3. Diffraction patterns recorded from a) ($CaSO_4$)surface region and b) ($CaCo_3$) deeper region. E=13 KeV

References

1) A.Cedola , V. Stanic , M.Burghammer , S. Lagomarsino, F. Rustichelli , R. Giardino , N. Nicoli Aldini, S. Di Fonzo, *X-ray micro-diffraction analysis of reconstructed bone at Zr prosthetic surface:characterization with sub-micrometer resolution of the interface between coating and bone*, Physics in Medicine and Biology, **48**, N37 (2003).

2) V. Komlev, A.Cedola , S. Lagomarsino, D. Rettura, M.Burghammer, V. Stanic , F. Rustichelli, R. Giardino, N. Nicoli Aldini, *Study of interface phenomena between bone and Zr coated and uncoated implants by microdiffraction*, to be published.