	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: 28 June 2001 to: 02 July 2001	**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 the first session, 28 June 2001 to 02 July 2001, which had mainly methodological objectives, has been used to show [1] the advantages, as compared to traditional X-ray diffraction, of using this innovative technique to study the interface between newly formed bone and a Zr prostheses with 0.5 micron spatial resolution.

The sample has been accurately scanned through the micro-beam and diffraction patterns have been acquired by the CCD. The careful analysis of the patterns allowed to extract the concentration profile of the different

crystalline elements coexisting in the interface region with a spatial resolution of 0.5 μ . From the analysis of the XRD pattern acquired at the interface region, the Zr ring broadening can be investigated, as shown in figure 1 for the [101] and [112] reflections. Figure 1 gives an indication of the structural variation of the Zr and suggests that in the interface region an important lattice deformation is present.

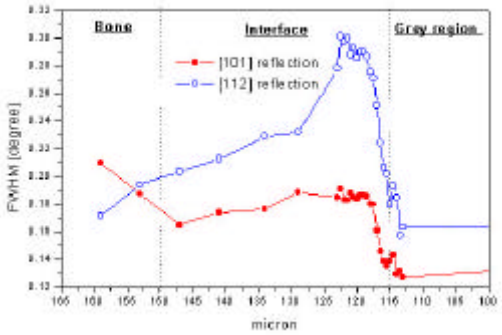

Figure 1. FWHM of the Zr peaks in the interface region where the bone starts to grow.

The measured diffraction spectrum of the reconstructed bone at the interface region, has been compared with the spectra of the trabecular and the cortical bone. A first qualitative comparison reveals that the pattern of the trabecular bone is characterised by the diffused diffraction rings indicating an important amorphous contribution likely coming from the collagen lattice.

Careful analysis of the spectra (see figure 2) reveals a marked difference between the new-formed and the native bone. The spectrum of the latter is perfectly compatible with the hexagonal system of the HA, as well known in literature while the spectrum of the new bone appears to be quite different.

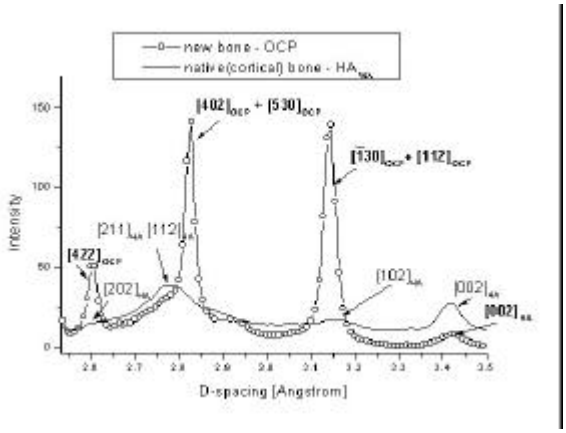

Figure 2. Normalised spectra of the HA measured for the reconstructed bone (straight line) and for the cortical native bone (dotted line). The two different crystallographic phases are evident.

From a detailed analysis it comes out that this experimental spectrum is compatible with the octocalcium phosphate (OCP) which can be a precursor in the bone mineralization. Indeed in the first step of bone formation the presence of the OCP phase should be substantial.

The power of this micro-diffraction technique can be clearly seen in figure 3 where the intensity variation of the reflections [130]+[112] of OCP at d=3.1454 Å and [002] of HA at d=3.4275 Å is studied at the interface region. The high spatial resolution reveals the abrupt transition from OCP to HA far from the Zr substrate and it confirms that the transformation of OCP in HA is proceeding towards the substrate.

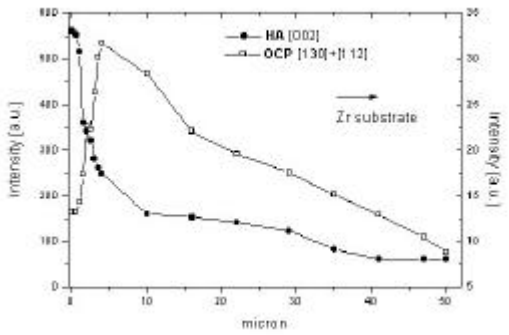

Figure 3. Intensity variation of the reflections [130]+[112] of OCP (open square) and [002] of HA (closed dot) at the interface region.

A detailed analysis of the ring profiles suggests that the grain size of the OCP is comparable with the beam size and larger then the grains of HA.

About the first session we can certainly conclude that these structural information can not be deduced by electron microscopy. In particular EDS measurements can only monitor Ca concentration, independently from its crystallographic phase. This limitation does not really allow to distinguish the newly formed bone from the native one.

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).