ESRF	**Experiment title:** Response of bone nanostructure and chemistry to bio-resorbable Mg implants	**Experiment number**: SC-3964
Beamline: ID21	**Date of experiment**: from: 24.09.2014 to: 28.09.2014	**Date of report**: 02.02.2015
Shifts: 9	**Local contact(s)**: Bernhard Hesse	*Received at ESRF:*

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Report:

Summary

Scanning X-ray fluoresence (XRF), x-ray near edge absorption spectroscopy (XANES) and infrared spectroscopy (FT-IR) have been used to probe the distribution and chemical state of Magnesium (Mg) and Phosphorus (P) in bones bearing degrading Mg implants of two different degradation speeds in a rat model (Sprague Dawley rat) over the course of implant degradation and bone remodelling (up to 18 months). The differences between the two Mg alloys and the zones of Mg accumulatin in the bone could be very well observed. The surrounding of blood vessles in the osteon structure is always a very favored place for Mg accumulation and acts as a reservoir for Mg in the bone. Whilst the degradation rates of Mg are high also the bone interface is suspected to high degrees of Mg accumulation. This effect levels out over time and the Mg content drops after total implant degradation to the normal level. These results are complemented by FT-IR which indicates changes in the structure of the collagen as suggested by an altered structure of the amide peak region together with the changes in the Phosphate regions during bone healing. Changes in the phosphate compounds are as well visible in the P-XANES spectra of selected points at the interface. Here a clear transition from the hydroxylapatite (HAP) structure, normaly present in bone towards an altered phase, possibly a diphosphate compound could be observed. However further investigation of this effect is necessary to get a conclusive picture.

Samples and Setup

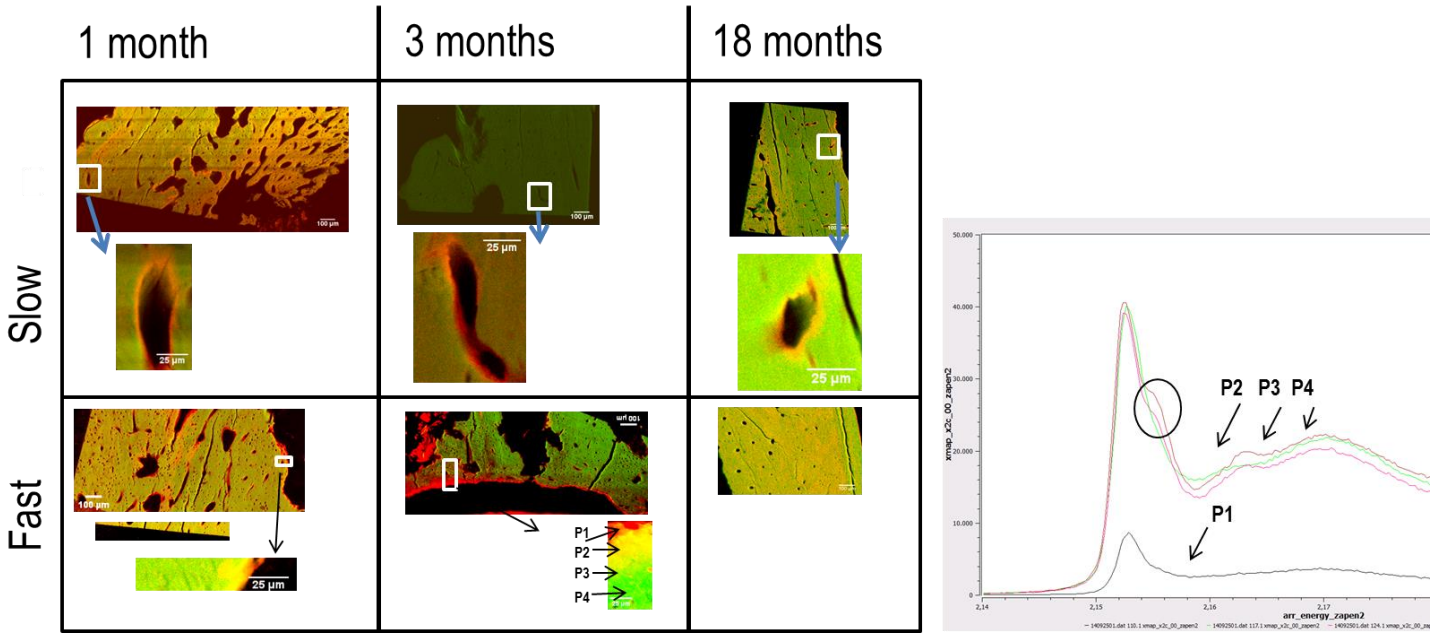
The XRF experiments were carried out at the SXM endstation. The energy for the fluoresence scans was set to 2150 eV, an energy still allowing to detect P without saturating the detector too much in terms of Mg detection. The samples were mounted 45° tilted with respect to the incoming beam and the detector and were measured in reflection geometry. The step size was choosen to be 3 µm for overview pictures and 0.5 µm for higher resolved details chosen from the overview picture. The beam was focused to a spot of 0.35x0.7 µm using KB mirrors. At selected points of the bone-implant interface with high Mg concentration XANES spectra have been measured in the energy range from 2140 to 2200 eV with an step width of 0.25eV.

The FT-IR experiments were carried out at the FT-IR endstation with an aperture setting of 8µm, the step rate was choosen as 4 µm. The samples scanned here were 1 µm thin slices of bone, mounted on TEM grids.
The aim of the investigation was to monitor the response of bone towards two medically relevant novel, degrading metallic implant materials, especially suited for surgical placement in children.
The samples comprised two sets of rat bones bearing two different Mg alloy types which show fast and slow degradation behaviour. For each of these alloys time points of 1,3 and 15 (fast)/18(slow) months were sampled with one sample from the implant interface and one 5 mm away from the implant. The here reported experimentes were complemented by SAXS/WAXS beamtime at ID 13 with the same samples. All the samples origin from the same region and are cut over a length of 10µm, ensuring good matching between the samples.

Principal outcome

The experiments yielded the following main outcomes:

- It was found that Mg is incorporated into the bone during implant degradation and the location of Mg concentration is dependent on the rate of Mg degradation. The Mg is not permanently incorporated into the bone structure but levels are reduced during bone remodelling in older samples.
- As shown by P-XANES, phosphorous binding is altered by the presence of Mg. Possibly a new mineral phase is formed.



The upper row of Fig 1 shows the slowly degrading implant. It can be seen that the Mg concentration is especially high in the surrounding of vessles. At the implant interface (lower side) no distinct Mg accumulation can be seen. After 18 months, a time point where the implant is gone for 3 months Mg can still be found in the surroundings of vessles.
The lower row shows the fast degrading implant type. Here a generally higher level of Mg can be seen. At the 3 months timepoint where implant degradation is the highest, Mg can as well be found in the implant interface of the bone. After 18 months, a timepoint where the implant is gone for 9 months, no elevated levels of Mg could be observed in the surroundings of the implant. P-XANES spectra at the points P1-P4 indicates a shift from the usual HAP structure at low Mg concentrations towards a different, possibly diphosphate structure in the zones of high Mg concentrations.

Conclusions and further proceedings

The results elucidate the presence and distribution of Mg and give an idea on how the Mg might affect the mineral structure of the bone. The results are highly relevant for the medical field, especially together with the results from SAXS and WAXS at the same samples and will substantially increase the knowledge on different mineral states induces by implant materials. A scientific publication is in preparation.
Proof of the possible existence of of new minteral phases will require further investigations of the local electronic state of P and possibly Mg at high Mg concentrations in bone.