	Experiment title: Response of bone nanostructure and chemistry to bio-resorbable Mg implants	Experiment number: SC-3964
Beamline: ID13	Date of experiment: from: 10.09.2014 to: 13.09.2014	Date of report: 02.02.2015 <i>Received at ESRF:</i>
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Report:

Summary

Scanning small-angle and wide-angle x-ray scattering(SAXS and WAXS) has been used to investigate the impact of degrading Magnesium implants and their degradation speed in a rat model (Sprague Dawley rat) over the course of implant degradation (up to 18 months). The nanostructure responded in general to the implant at the interface by a realignment of mineral platelets and an altered mineral platelet thickness. Furthermore the lattice of the hydroxylapatite (HAP) mineral is locally altered. Together with the corresponding experiments at ID21 it could be shown that it is the presence of elevated Mg levels that induces the observed changes in the nanostructure of bone as well as the mineral structure of HAP.

Samples and Setup

All experiments were carried out on the microfocus branch of ID13 using a set of compound refractive lenses to achieve a spotsize of 2.5x5 µm at an energy of 12.5 keV(SAXS) and 13.6 keV (WAXS) respectively. Scattering images were recorded using a FReLoN 4M camera. In order to monitor the mineral content at the probed spot, a VORTEX-EM fluorescence detector was additionally mounted to detect predominately the Ca K_α emitted by the HAP mineral.

The aim of the investigation was to monitor the response of bone towards two novel medically relevant 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 samples were embedded in PMMA resin and cut to a slice thickness of 1 µm with subsequent sample mounting on TEM grids.

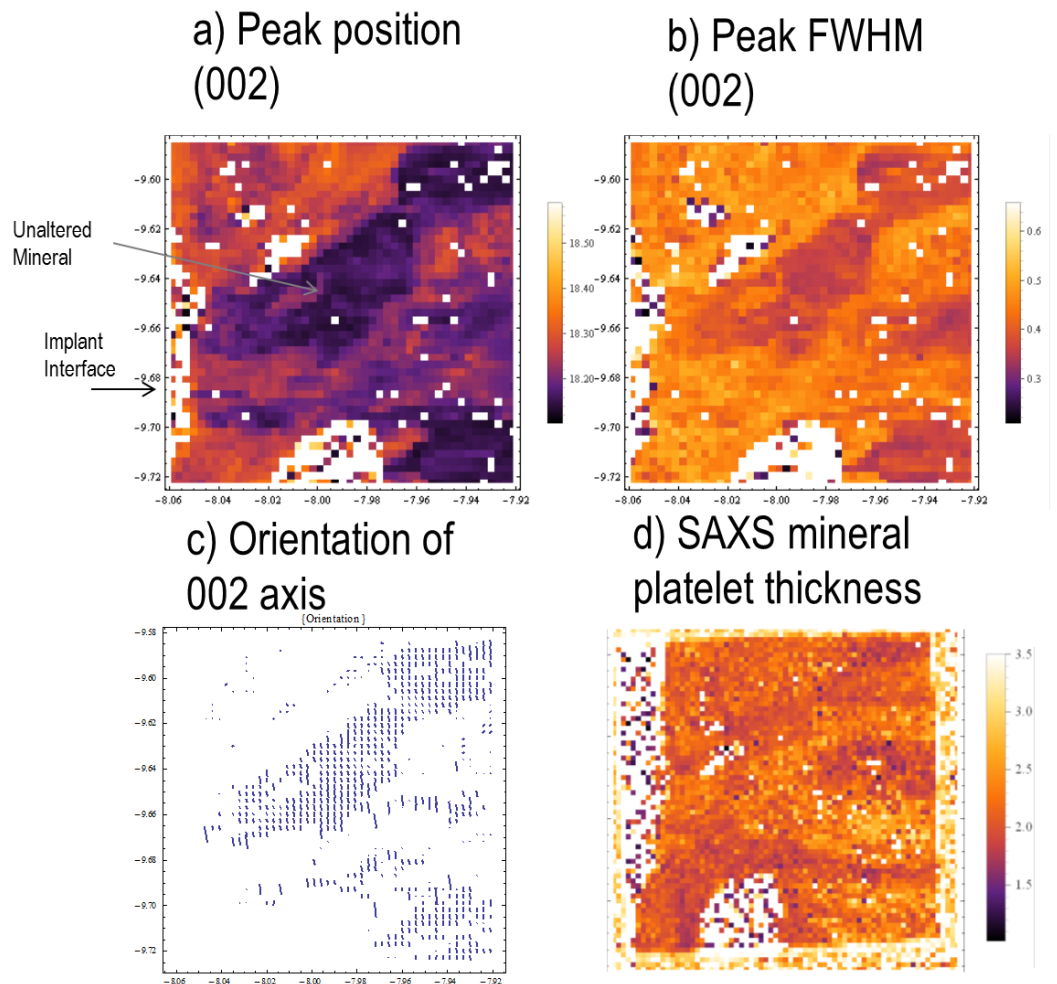
Thanks to the high flux and the correspondingly small acquisition time and the optimized experimental setup at ID13 we have been able to sample areas as large as 200x200 μm , allowing to separate morphological features from implant induced effects.

Principal outcome

The experiments allowed to verify two important hypotheses.

- The bone nanostructure responds to the presence of an degrading Mg implant by nanostructural realignment of the bone mineral matrix
- The bone mineral structure is altered in zones of high Mg levels. A difference is visible between the two implant types and their respective rate of Mg release.

Fig. 1 shows the bone implant interface exposed to the strongest Mg concentration level. The position of the 002 reflection (Fig 1a) is very good indicator on lattice distortion as Mg induces a shift towards higher q . A zone of unaltered mineral can be seen, surrounded by mineral that is obviously affected by the presence of Mg. This presence coincides with an increased Scherrer widths, indicative for smaller crystallites as shown in Fig1 b). Another remarkable observation is the abrupt loss of orientation outside the zone of unaltered HAP mineral depicted in Fig 1c). These changes in the bone mineral structure are as well present on the bone nanostructural level as probed by SAXS. Fig 1d) shows the platelet thickness of the mineral particles as extracted from the SAXS measurements by a stack of cards model. Here is also an increased mineral platelet size in zones of unaltered mineral visible.



Conclusions and further proceedings

From these results together with the complementing XRF experiment on the same samples at ID21 we could show that the degrading Mg implant has an impact on the mineral- and nanostructure of bone during bone remodelling. The influence of local Mg levels on bone mineral formation is a crucial factor for the healing process and bone stability. Together with our medical cooperation partners at MedUniWien, these results will be used for further optimization of implant alloys. A scientific publication is in preparation.