	Experiment title: Spatial distribution of magnesium around biodegradable implants	**Experiment number**: MA 3043
Beamline: ID 20	**Date of experiment**: from: 4.6.2017 to: 7.6.2017	**Date of report**: 31.5.2017
Shifts: 9	**Local contact(s)**: Christoph Sahle	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

D.C.F. Wieland*, Helmholtz Zentrum Geesthacht, Max-Planck-Straße 1, 22761 Geesthacht

Silvia Galli*, Laboratory Faculty of Odontology Malmö Univeristy Carl Gustavs Väg 34 SE - 20506 MALMO

Christoph Sahle*, Laboratory ESRF 71 avenue des Martyrs CS 40220 FR - 38043 Grenoble Cedex 9

Imke Greving*, Helmholtz Zentrum Geesthacht, Max-Planck-Straße 1, 22761 Geesthacht

Regine Willumeit-Römer , Helmholtz Zentrum Geesthacht, Max-Planck-Straße 1, 22761 Geesthacht

Report:

We investigated the magnesium distribution around biodegradable model implants made of magnesium alloys studied by a tomography approch which is based on x-ray Raman scattering (XRS). Magnesium is thought to be a good candidate for biodegradable implants but its incorporation into bone and its degradation process are not fully understood. Histological studies showed that the bone formation is altered in the surrounding of a magnesium implant. [1, 2] For a better understanding of the corrosion process and a knowledge based development of magnesium implants, the distribution and chemical state of magnesium in the bone matrix is of great interest. In conventional elemental analysis investigations, the specimen has to be cut in slices for investigations because of the low x-ray energies. [1] This drawback can be overcome by direct tomography (DT). [4] By connecting the elemental distribution gained from DT with high resolution μCT measurements of the same specimen a better understanding of the mechanism will be gained, as the magnesium distribution and oxidation state can be directly connected to the structural changes in the bone matrix.

For the measurements we used critical point dried samples of a piece of rat femur with an embedded magnesium implant. The bioresobable screw was made of the magnesium alloy Mg10Gd. The investigated healing period was 12 weeks. Measurements were performed at

the ID20 with an energy of 10keV and 13keV in order to test which energy was better suitable to maximise the signal with respect to absorption, energy resolution and scan-time. From the measurements it turned out that an energy of 13keV was most suitable as the pentration depth was increased and bsorption was reduced. The measuremens were performed at the elastic line as well as around the magnesium L-edge. Further the super resulution alogortihm was applied on the data, which increased the counts due to combining the different dectrors and enables us to reduce the scanning time in further experiments. [5]
Figure 1 shows the intensity of the elastic line with a resolution of 50µm along with a conventional µCT image collected on tomography labsource of the sample. A good correspndance between the samples can be seen.

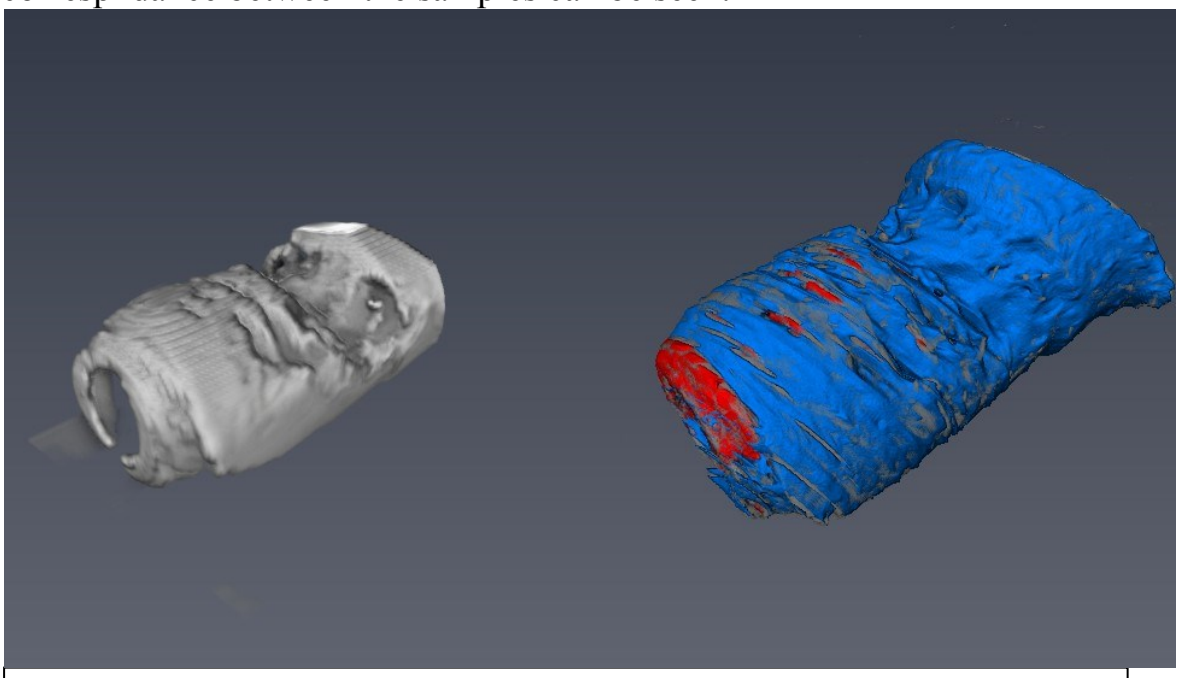

Figure 1: Left. Three dimesional distribution of the signal from the elastic line. Right.: Segmented tomogram from the sample. The Mg Screw (red) and Bone (blue) are segmented for better visibilty.

As the sample was quite large 4mm x 4mm x 6 mm, not the complete sample was scanned with highest resolition. Only a small local area of the size 500 µm x 500µm was scanned with the highest resolution resultuing in a voxel size of 10µm x 10µm x 1 µm. Figure 3 shows this high resolution scan of the implant sample in grey colours.
Figure 2 shows the Magnesium L-edge of the bone sample at 49eV which was chosen to perfrom our studies. The spectra show a very well defined white line and, thus, overs the opportunity for the chemical bond contrast.
For this, scans before and at the magnesium L-edge were taken. The difference between them was calculated and used as a contrast. Figure 3 shows this contrast difference of the edge along with the high resolution scan.

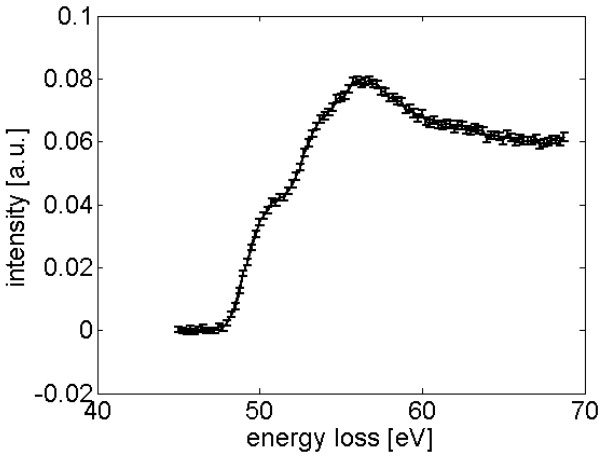

Figure 2 Magnesium L-edge measured in the bone sample with the Mg5Gd screw.

From this figure the magensium distribution in the bone can be nicley seen. And it shows the feasibilty of the technqiue to study the Mg

distribution in bone samples. However, some further analysis and data threatment has to be performed in order to make qualitative statements about the Mg distribution. In the ongoing data analysis two major points have still to be considered. First the dataset has to be accounted for absorption and second the µCT image and and the XRS-image have to be overlayed to draw combined conclusions from the chemical and strucutral information. For this both datasets will be registered and spatially correlated. Here problmes still exist due to the different spatial resolution of the sample and due to the incompletness of the XRS-image (shadowing of the backpart). After this is accomplished, also the data can be corrected for absorption. Here we will use the µCT data and the information from the elastic line as both datasets contain equal information with respect to the density of the material.

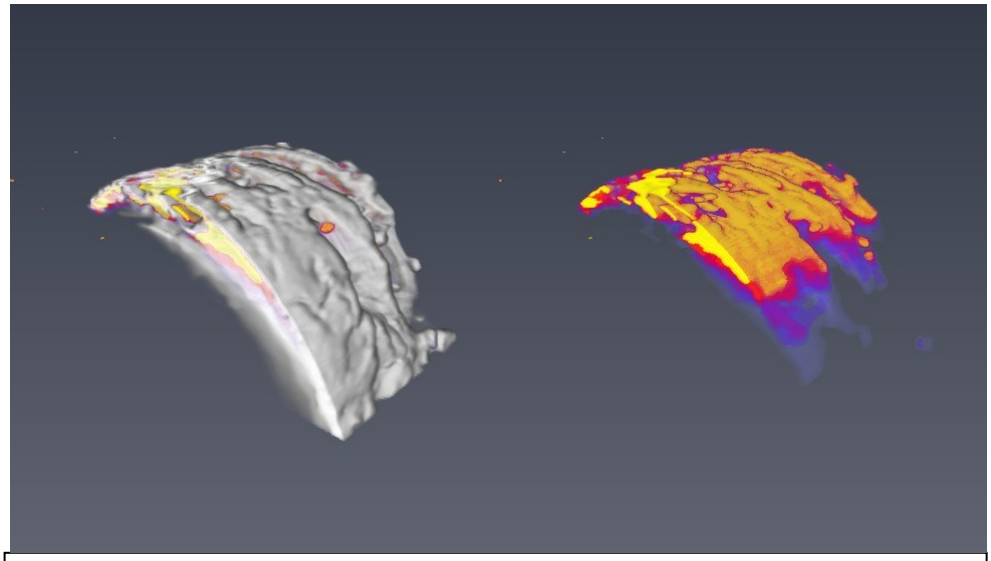

Figure 3, Left) High resolution scan of a part of the implant with bone.Right) Diffrence of the pre-edge and post-edge, which is also overlayed in the left image.

1. Witte, F., et al., *In vivo corrosion of four magnesium alloys and the associated bone response.* Biomaterials, 2005. **26**(17): p. 3557-3563.
2. Witte, F., et al., *Biodegradable magnesium scaffolds: Part II: Peri-implant bone remodeling.* Journal of Biomedical Materials Research Part A, 2007. **81A**(3): p. 757-765.
3. Reifenrath, J. et al. *Magnesium alloys as promising degradable implant materials in orthopedic research,* Magnesium Alloys Corrosion and Surface Treatments, 2011, p. 93 -108
4. Huotari, S. Direct tomography with chemical-bond contrast, Nature, 2011, **10**, p. 489-493
5. Sahle, Ch J., et al., "Improving the spatial and statistical accuracy in X-ray Raman scattering based direct tomography." *Journal of Synchrotron Radiation* **24**, no. 2 (2017): 476-481.