| | Experiment title: Investigating distribution and speciation of micro- and nanoparticles surrounding invivo ceramic and titanium dental implants | Experiment number: MD1115 |
|---|---|---|
| **Beamline**: ID21/ID16B | **Date of experiment**: 16/07/17-21/07/17 (ID21) and 01/09/17-04/09/17 (ID16B) | **Date of report**: 23.02.2018 |
| **Shifts:** 18 | **Local contact(s)**: Vanessa Suarez (ID16B) Bernhard Hesse (ID21) | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

Tobias Fretwurst * (University Medical Center Freiburg, Hugstetter Straße 49, 79106 Freiburg)

Katja Nelson (University Medical Center Freiburg, Hugstetter Straße 49, 79106 Freiburg)

Bernhard Hesse * (European Synchrotron Radiation Facility (ESRF), 38043 Grenoble Cedex 9, France)

## Background

Every year, 12 million dental implant are placed worldwide (in Germany approximately 800.000/year) [1]. Up to 47 % of these implants develop an inflammation of the surrounding tissue [2]. These inflammations require further medical invention with negative impacts on life quality and high cost for the health care system. Titanium based alloys are the most common dental implant types. The role of implant derived Ti nano-particles (NP) and especially the role of the Ti-NP size, Ti distribution (intra- vs extracellular) and speciation (anatase vs rutile) were still not clear during the course of inflammation [3]. Recently also ceramic based dental implants which provide very good mechanical properties and low failure rates [4] are inserted, commonly made of Yttria stabilised tetragonal zirconia polycrystalline (YTZP). During our experiment we investigated the dental implant derived particle distribution in the peri-implant tissue that showed inflammation. The elemental information, distribution and size of expected NP or small congregations containing Al, Ti, V and YTZP has been investigated at ID16B and ID21. In addition Ti speciation has been investigated at ID21.

## Experimental setup

**ID21:** Beam size was about 0.4 µm (vertical) x 0.9 µm size (horizontal). Energy for ceramic samples was 2.4 keV (plus a higher harmonic at 7.2keV), for the Ti-containing samples we worked at an energy of 5.1 keV.We have scanned a total of 15 samples of peri-implant tissue with inflammation extracted from different patients, 7 of which have a Ti implant and 8 have a YTZP implant. We have collected overview maps at between 5 µm and 25µm resolution (fig. 1 B and C) to identify ROI's for further investigations at 2 µm, 1 µm, and 0.5 µm resolution (fig. 1 D and E). Furthermore we have collected XANES for Ti particles.

The preliminary data analysis suggests:

- Presence of both Ti and YTZP particles in peri-implant regions of sizes ranging from below 1µm to up to 10 µm
- Ti particles speciation, derived by XANES, revealed different $TiO_2$ structures, one example is shown in fig 1 F, a detailed analyses is ongoing.

**ID16B:** Beam size was 60 nm x 60 nm, energy was 29.6 keV. We have scanned a total of 7 samples of peri-implant tissue with inflammation extracted from different patients, 3 of which have a Ti implant and 4 have a YTZP implant.

These samples had already been scanned during the ID21 experiment, and we investigated the same ROI's. We collected measurement with a resolution of 2 µm and 1 µm in order to identify their location, and then collected precise scan with a resolution of 60 nm (fig. 1 G).

The preliminary data suggests :

- What appeared as a single particle of µm size at ID21 is actually a cluster of smaller NP which can be smaller than 60 nm
- The co-localization of Zr and Y in individual particles confirms the provenance from the dental implant

## Conclusions/discussion

The beamtime went technically extremely well and the results are very promising. This study confirms the complementarity of the two beamlines ID21 and ID16B for this kind of samples. We detected Ti NP in all 8 samples of tissue surrounding Ti implants, and YTZP NP in 5 of the 8 samples of tissue surrounding YTZP implants. These experiments confirm the release of Ti NP around dental implants, and, to our knowledge, this is the first study addressing the release of YTZP NP around dental implants. Currently, immunohistological tests matching the fluorescence maps are ongoing to pinpoint immune cells in particle-containing regions. The precise information about Ti particles speciation opens possibilities for better toxicology tests. A publication for a high impact journal is in preparation.

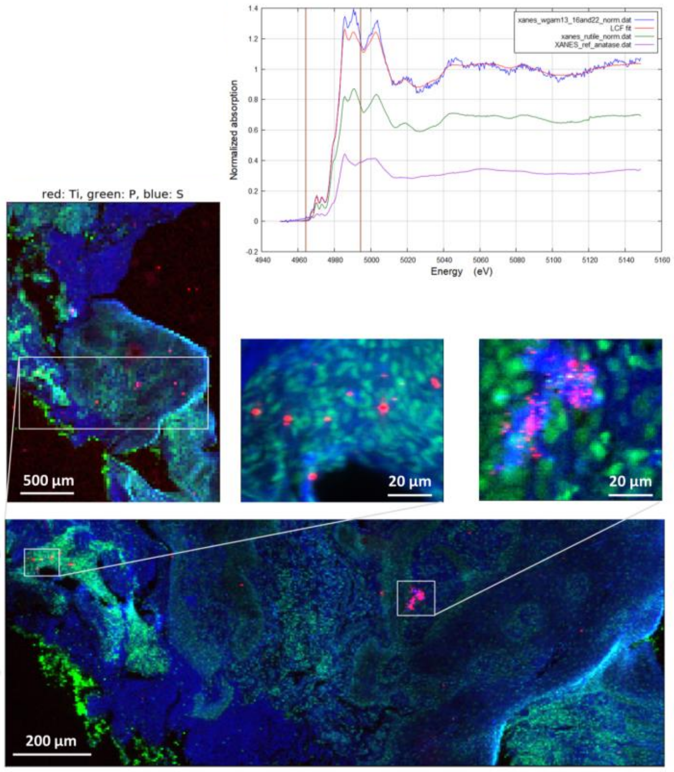
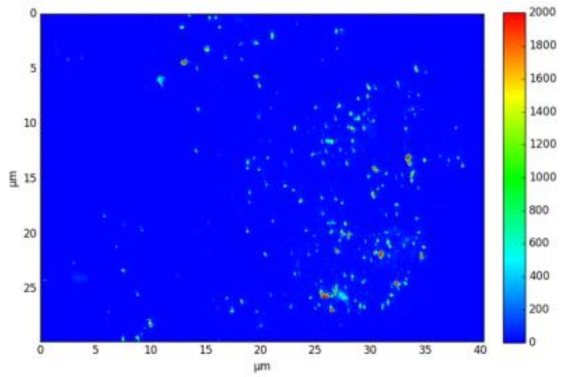



Figure 1 (left): ID21: Distribution of Ti-NP in a peri-implant tissue. Linear combination fitting revealed that the analysed particles of this sample is composed of ~70% rutile and

Figure 2 (below): Distribution of Ti-NP revealed by nanoXRF on beamline id16B at 60 nm beamsize.




[1] Albrektsson et al.. Is marginal bone loss around oral implants the result of a provoked foreign body reaction? Clin Implant Dent Relat Res. 2014;

[2] Derks J &Tomasi C. Peri-implant health and disease. A systematic review of current epidemiology. Journal of clinical periodontology. 2015

[3] Skocaj et al.; Titanium dioxide in our everyday life; is it safe? Radiology and Oncology,2011

[4] Spies et al.; Evaluation of Zirconia-Based All-Ceramic Single Crowns and Fixed Dental Prosthesis on Zirconia Implants: 5-Year Results of a Prospective Cohort Study. Clin Implant Dent Relat Res. 2015