Synchrotron techniques for materials characterization

Berit Zeller-Plumhoff

Data-Driven Analysis and Design of Materials
Universität Rostock

What is your background?

Why are you joining the lecture?

Aim of the lecture:

You will be familiar with synchrotron radiation-based **techniques** used for materials characterization and understand their **underlying principles**. Moreover, you can assess the **requirements** for a successful measurement and would be able to **plan and analyse** an experiment given a certain question.

About us

Berit Zeller-Plumhoff

TUM — BSc. + MSc. Mathematics

University of Southampton — PhD Materials Science/ Bioengineering

Nikon — Industrial secondment

Helmholtz-Zentrum hereon — Postdoc Head of Department

ZEISS — Application Engineer

since 01.08.2024 professorship CDMA

Carsten Wickmann

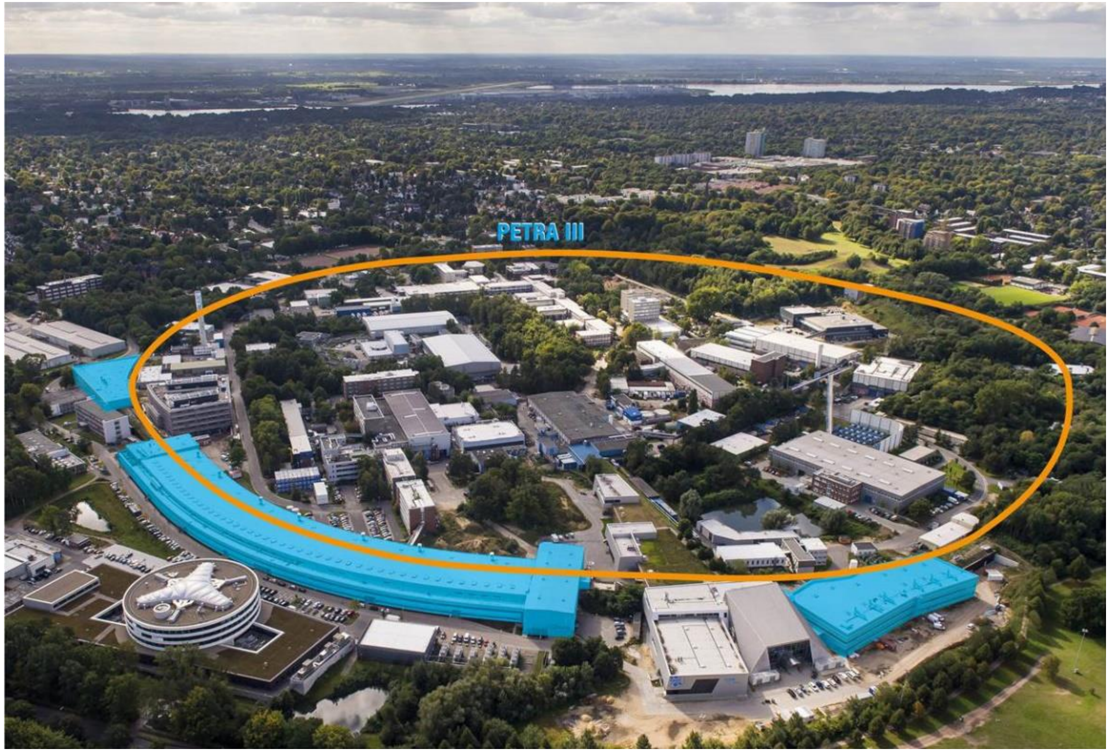
Universität Rostock Traditio et Innovatio — B. Sc. + M. Sc. + IWE mechanical engineering

Universität Rostock Traditio et Innovatio — PhD structure mechanics, fatigue of materials

Fraunhofer IGP — Research assistant fatigue of weldments, numerical simulations, fotogrammetry

since 01.10.2024 research assistant CDMA

Motivation



http://photon-science.desy.de/facilities/petra_iii/index_eng.html

Do you have experience in X-ray techniques and image processing?

What are you interested in?

About the lecture

- 2 SWS Lecture (english) – R115: Fundamentals and theory
- 2 SWS Tutorial (english) – R109 (PC-Pool): Programming with Jupyter Notebooks to perform calculations, generate graphs and perform image processing
- Excursion to DESY in January 2025
- Inverted classroom for last 30%

- Exam: written exam of 120 minutes

- If you have questions, comments or feedback, please contact us:

  berit.zeller-plumhoff@uni-rostock.de, carsten.wickmann@uni-rostock.de

Lecture content

Date	Lecture content	Comment
15.10.2024	Introduction and overview	
23.10.2024	Generation, interaction and detection of X-rays	
30.10.2024	X-ray computed tomography	
06.11.2024	X-ray computed tomography	Start at 9:00 am sharp
13.11.2024	Propagation-based phase contrast	
20.11.2024	X-ray microscopy	need to move date
27.11.2024	cancelled	
04.12.2024	X-ray diffraction - Small angle X-ray scattering	
11.12.2024	X-ray absorption and fluorescence spectroscopy	
18.12.2024	Image processing	

Lecture content

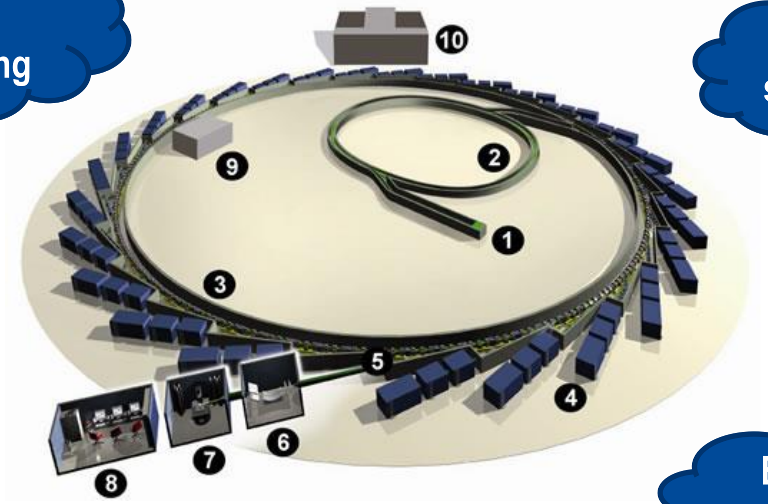
Date	Lecture content	Comment
08.01.2025	DESY excursion	
15.01.2025	Image processing	
22.01.2025	Image processing	
29.01.2025	Outlook: neutrons and exam preparation and questions	

Literatur

- Literature: J. Als-Nielsen, Elements of Modern X-ray Physics, 2nd Ed., Kaptl. 9
- A number of papers that will be made available via StudIP

Synchrotron techniques for materials characterizeration

3D Imaging



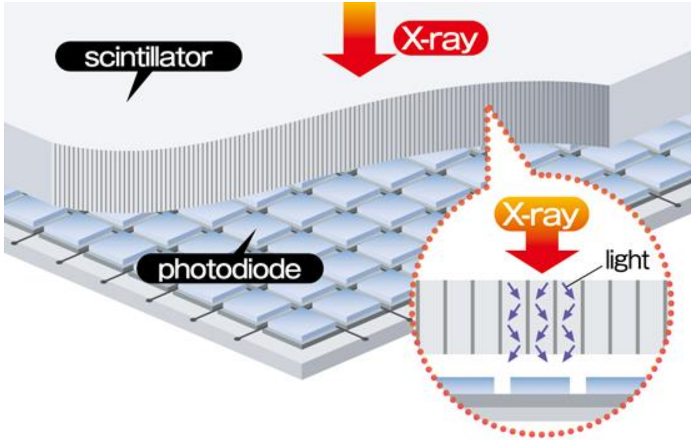
Crystal structure

Elemental composition

https://www.diamond.ac.uk/Science/Machine/Components.html

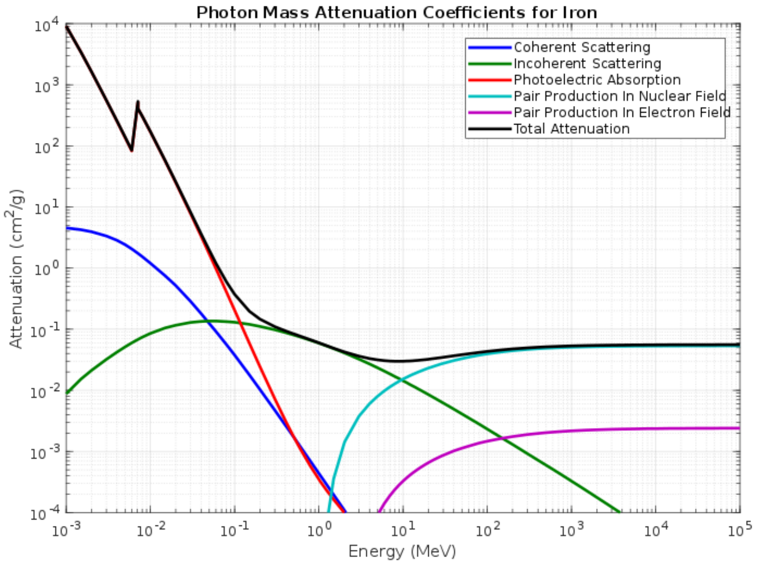
X-ray generation and detection

http://photon-science.desy.de/facilities/flash/the_free_electron_laser/ undulator/index_eng.html



https://www.konicaminolta.com/healthcare/products/dr/dr30/index.html

Interaction of X-rays with matter



https://en.wikipedia.org/wiki/Mass_attenuation_coefficient

X-ray computed tomography

from www.shutterstock.com

X-ray computed tomography
Phase contrast imaging

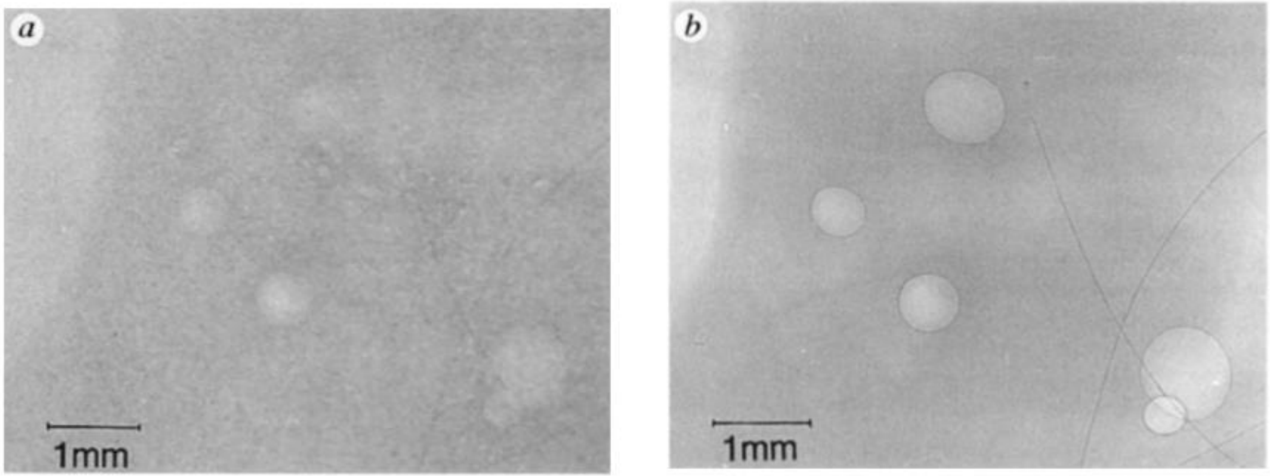
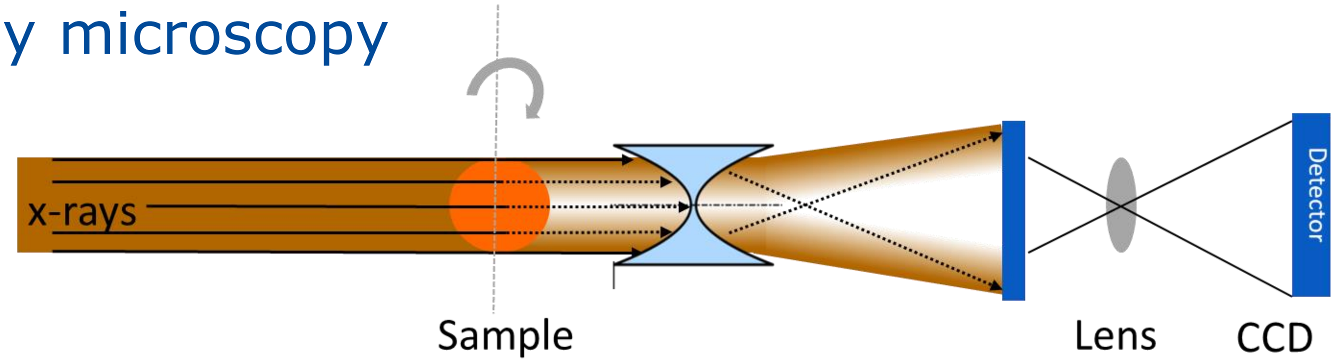


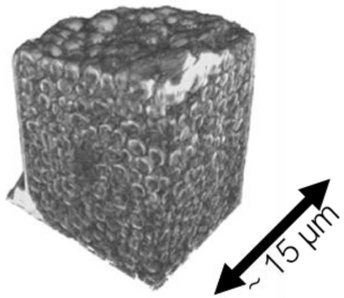
FIG. 3 Images of air bubbles and glass fibres in a polymer glue ('Tarzan's grip', Tarzan's Grip Products, Milperra, NSW, Australia). This is a similar sample to that reported in ref. 8 and corresponds to an almost pure phase object. Source–object distance R_1 was 200 mm, and object–image distances were $R_2 = 1$ mm (panel *a*; 15-s exposure) and 1,200 mm (panel *b*: 8-min exposure). The tube voltage used was 60 kV. Image *b* shows black/white contrast at edges of bubbles and also at edges of fibres, corresponding to additional contrast over that expected for a normal absorption contrast image (*a*).

Taken from Wilkins et al., Phase-contrast imaging using polychromatic hard X-rays, Nature , volume 384, pages 335–338 (1996)

X-ray microscopy



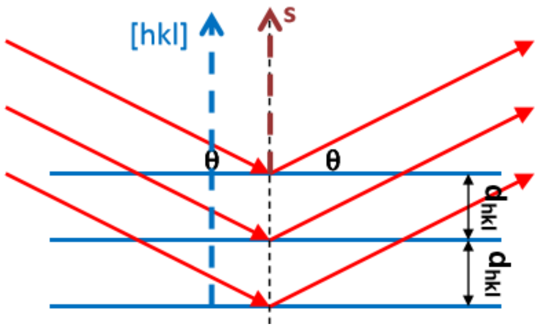
Nominal line width 50 .... 200 nm

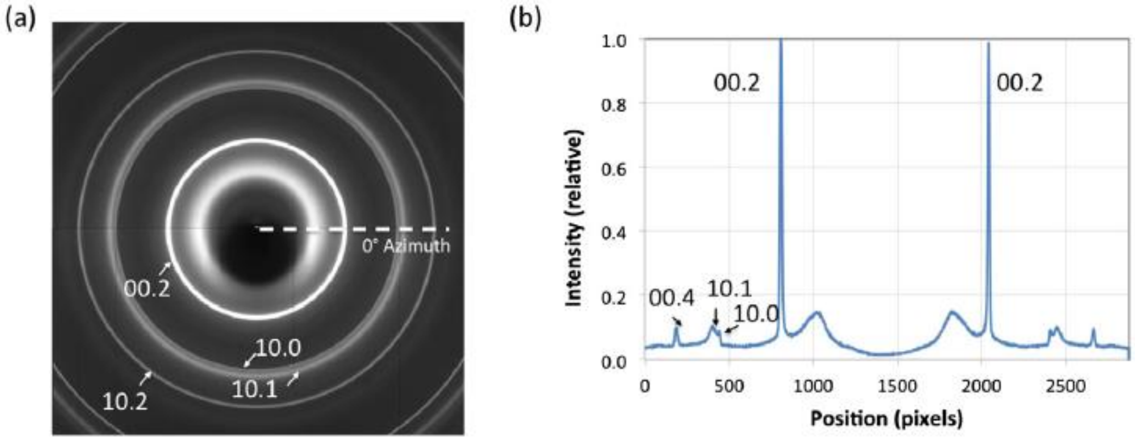


Visualized 3D volume of photonic glass sample; bead diameter ~2 µm

Slide courtesy of Dr. Imke Greving

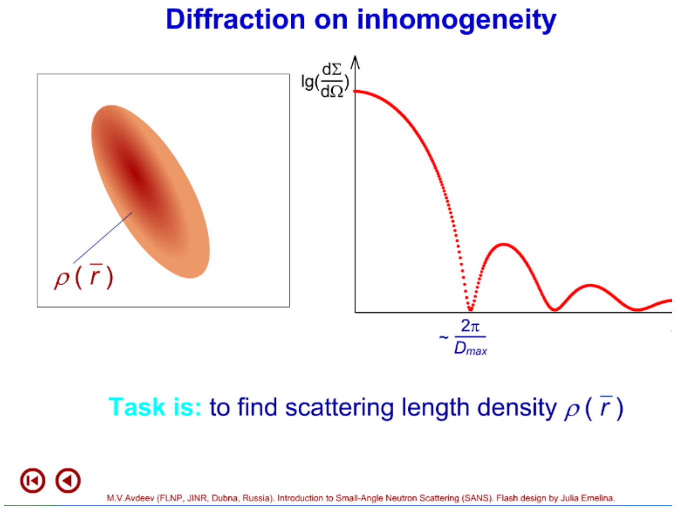
X-ray diffraction

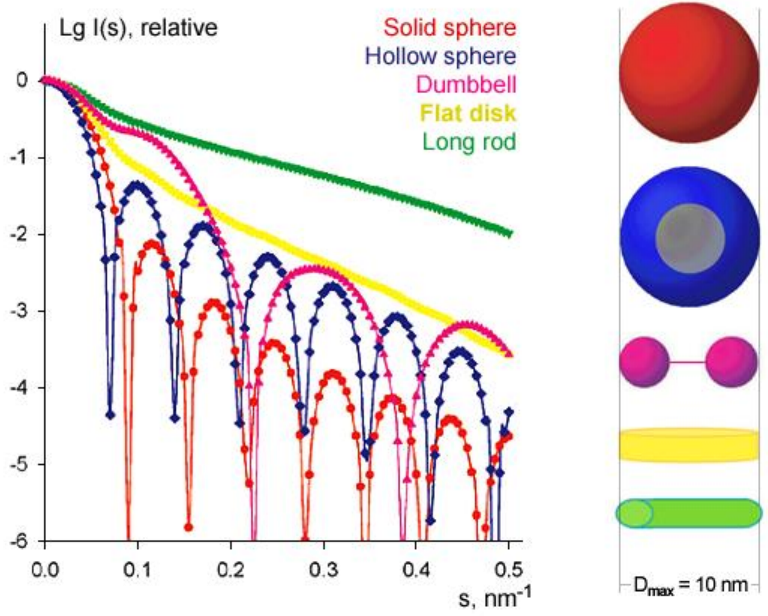




Taken from Marrow et al. (2016) https://doi.org/10.1016/j.carbon.2015.09.058

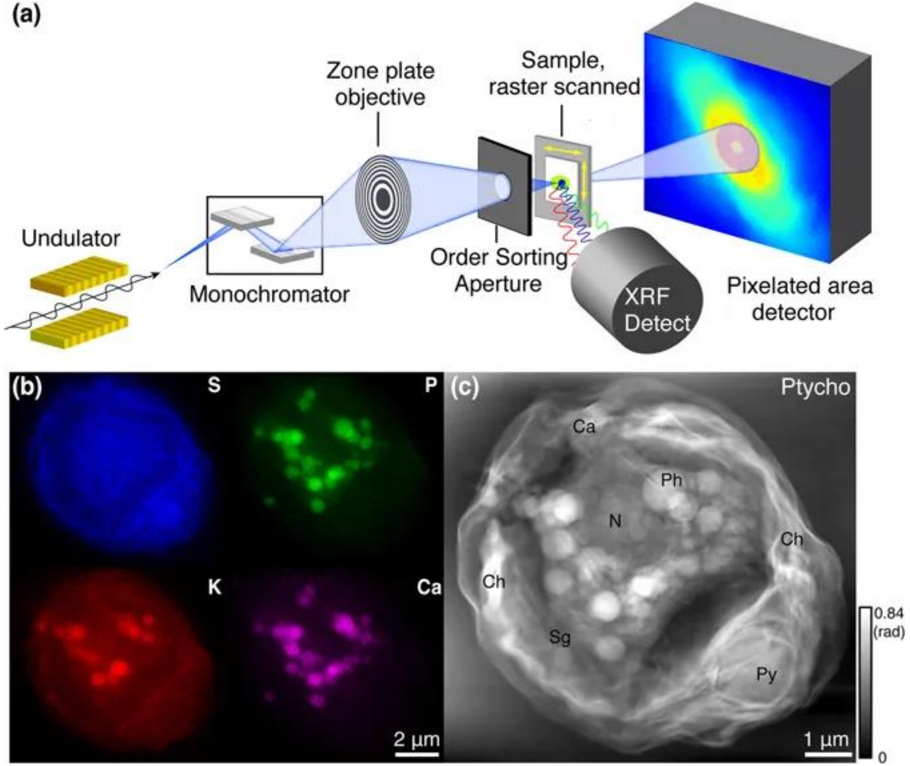
Small angle X-ray scattering





Svergun & Koch: Rep. Prog. Phys. 66 (2003) 1735–1782

X-ray fluorescence



Taken from Deng J. et al., Scientific Reports 7, 445 (2017)

Image processing

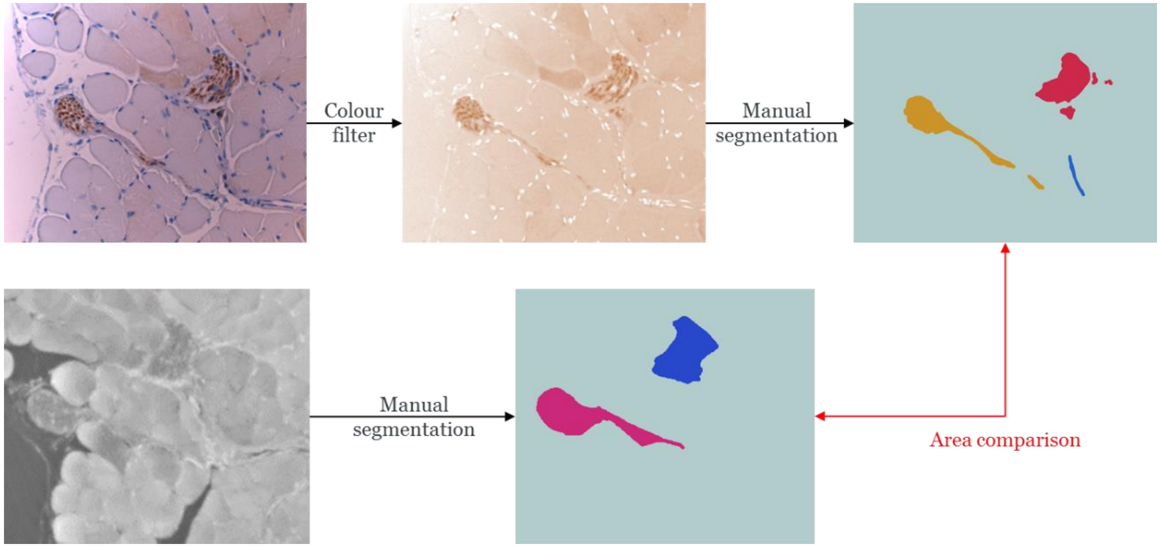
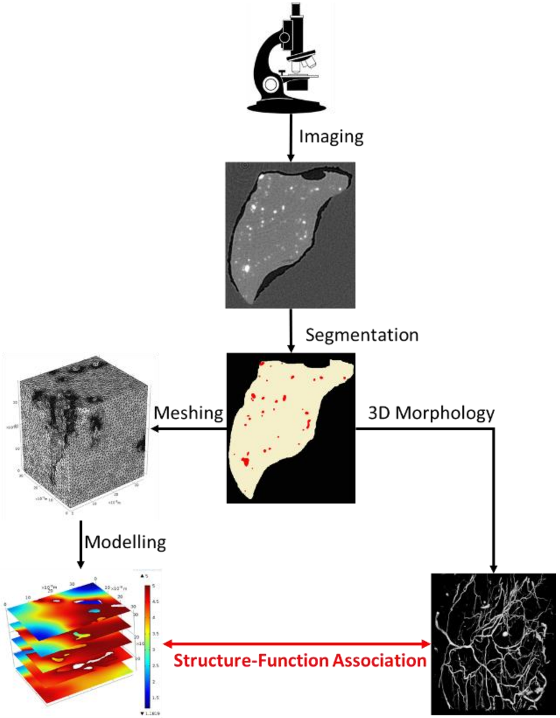
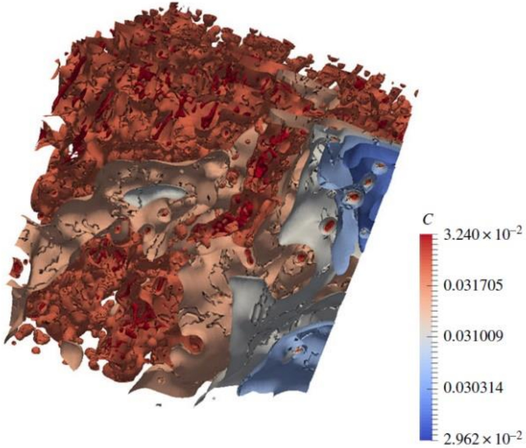


Image-based modelling

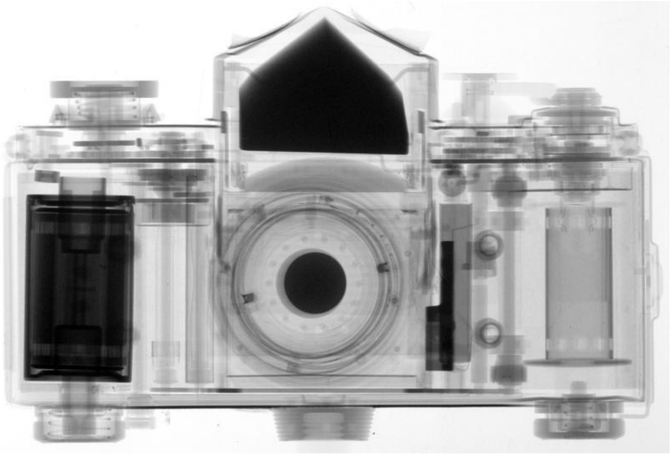


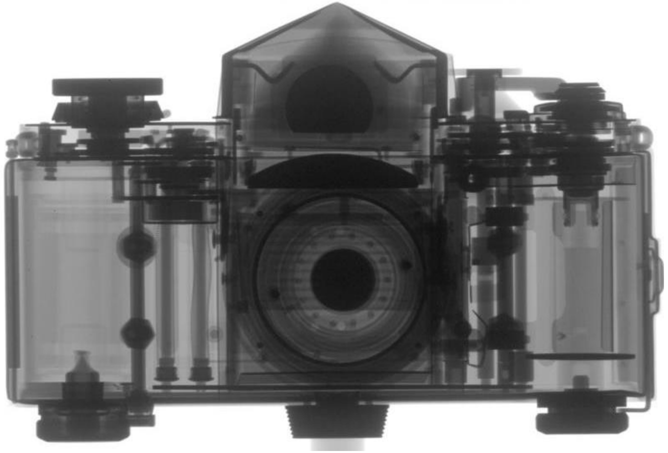
Zeller-Plumhoff B, et al. 2017. J. R. Soc. Interface 14: 20160992.



Zeller-Plumhoff B, et al. 2017. J. R. Soc. Interface 14: 20170635.

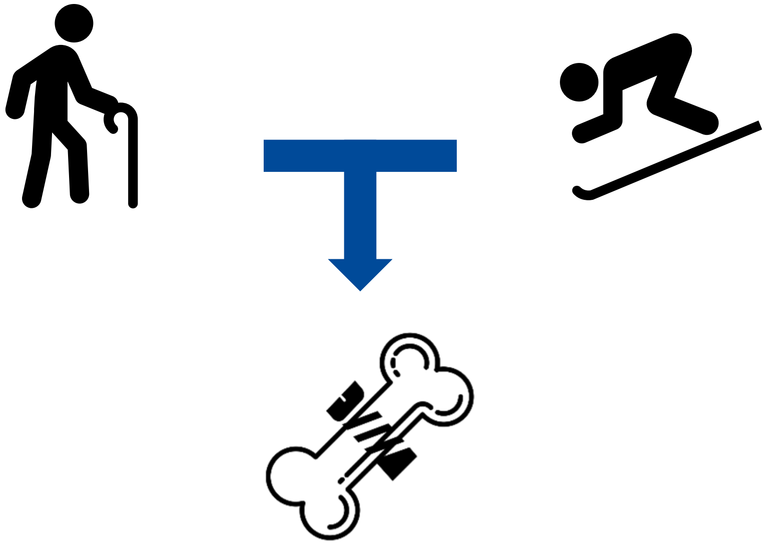
Outlook: neutron techniques





https://www.psi.ch/de/niag/what-is-neutron-imaging

Motivation: Magnesium-based implants



Global impant market 2019: several billion €

Magnesium-based implants

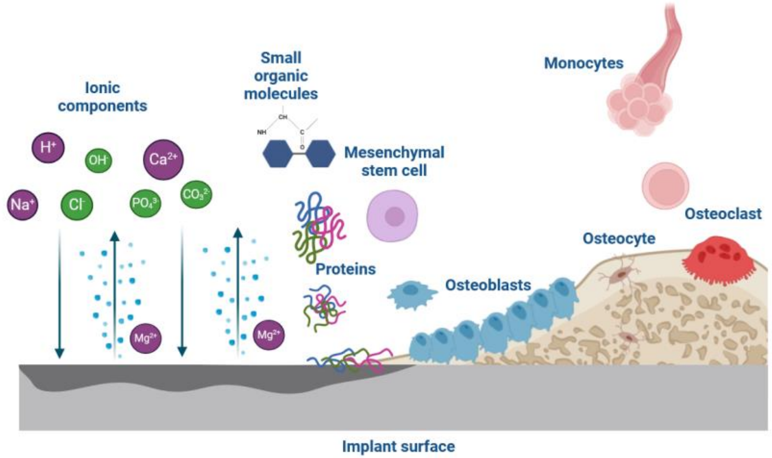
- Biocompatibility

- Biodegradability
  - Temporal support of bone
  - Local change of chemical environment

➢ Influence of ionic components

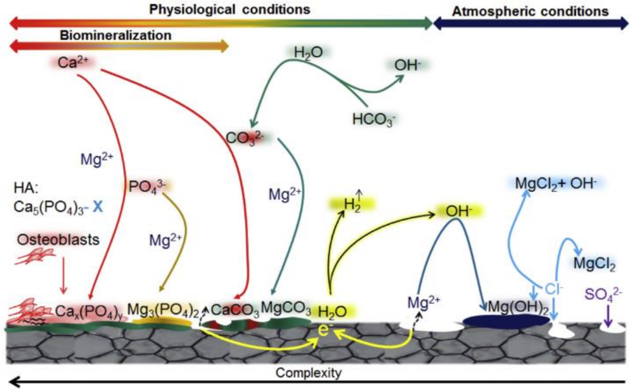
➢ Influence of proteins and cells

➢ Influence of intermetallic phases and impurities

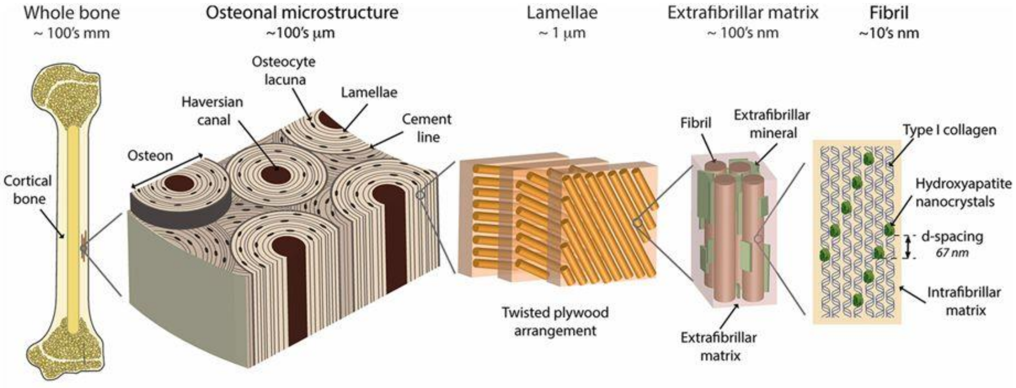


➢ Development of a predictive model of magnesium biodegradation and the tissue response

Investigating biodegradable bone implants



J. Gonzalez et al., Bioactive Materials (2018)



Zimmermann et al., Scientific Reports (2016)

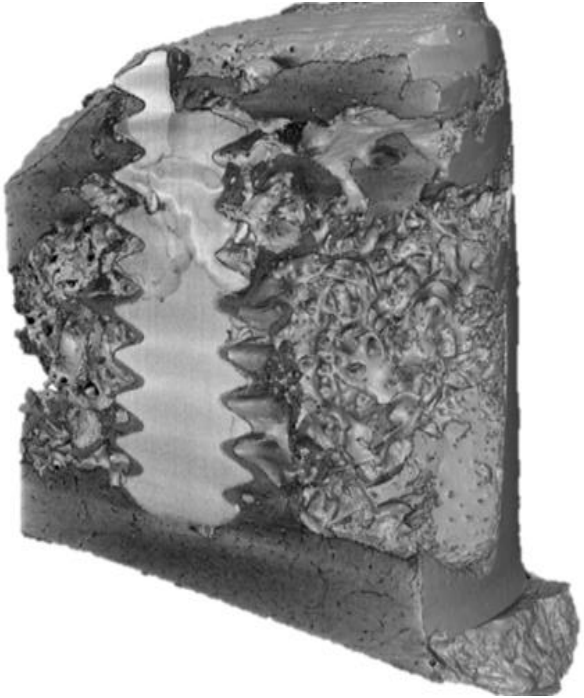
Investigating biodegradable bone implants

Implant:

- Degradation
  - Morphology
  - Structure
  - Chemical composition

Bone

- Growth
  - Morphology
  - Ultrastructure
  - Chemical composition
  - Biomechanical properties



Experimental design

Diana Krüger Hanna Ćwieka

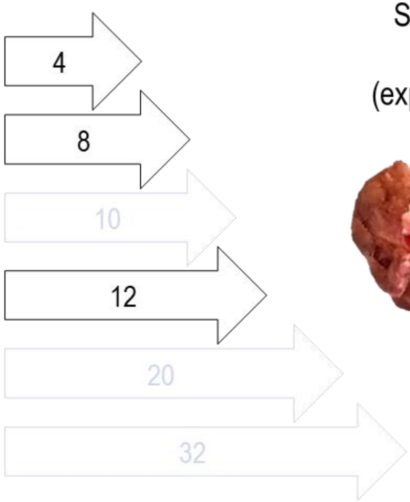
Materials:
- Mg-5Gd
- Mg-10Gd
- Titanium

M2 screw

@Hereon

Sprague Dawley

@Malmö University, Malmö, Sweden
@MOIN CC, Kiel, Germany



Healing time [weeks]

Example: Mg-5Gd screw in Sprague Dawley rats tibia (explant size: ≈ 6x6mm)

Röntgen Ångström Cluster

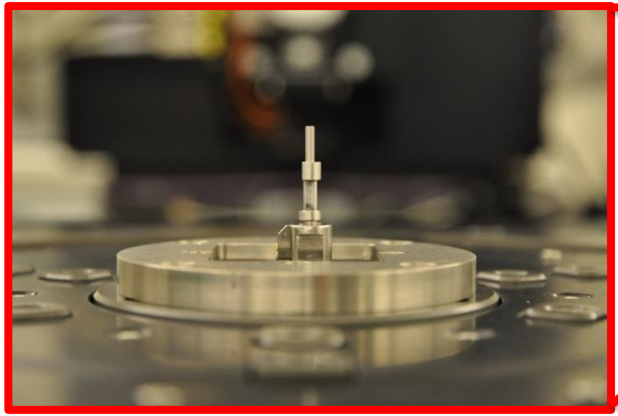
Bundesministerium für Bildung und Forschung

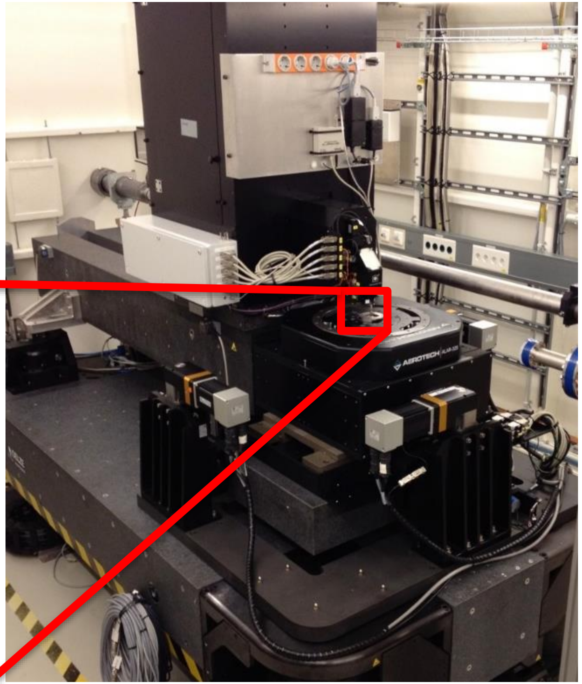
Krüger et al., Bioactive Materials (2021)

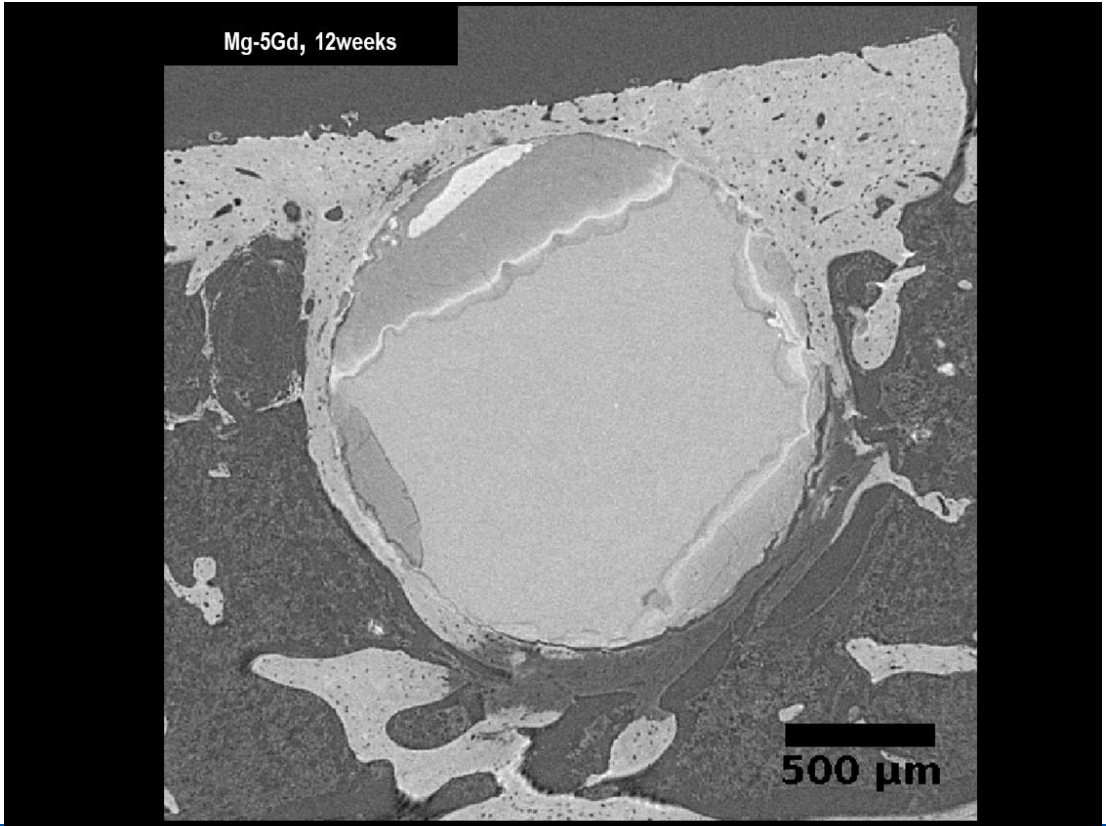
High-resolution computed tomography P05 beamline at PETRA III

Materials Design and Characterization

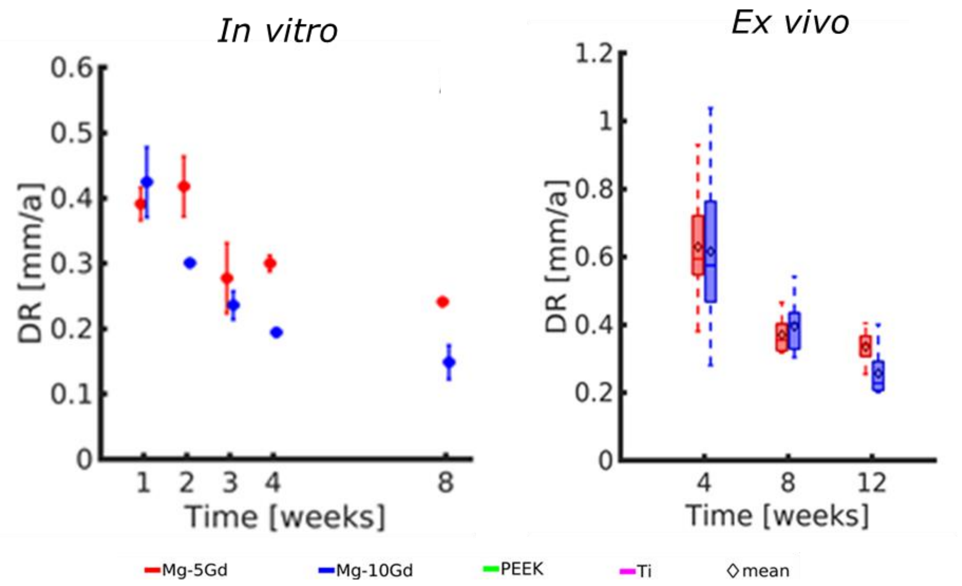
- FOV: 7.4 x 2mm
- Resolution < 1µm
- Additional space for *in situ* experiments and sample environments

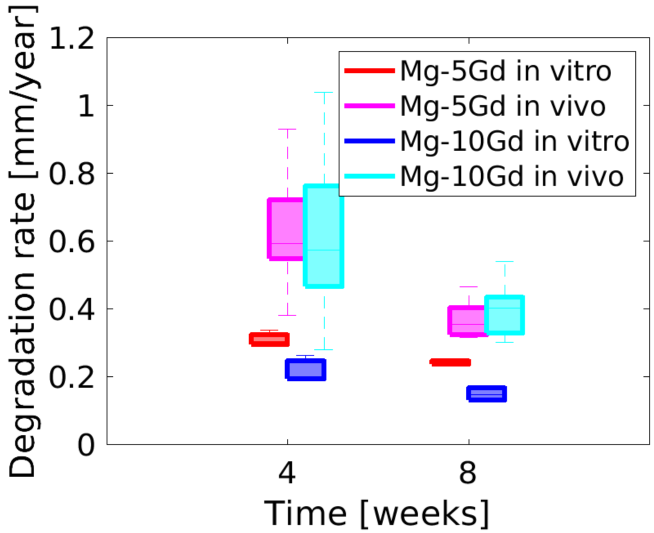





Mg-5Gd, 12weeks
500 µm

Degradation rates *in vitro vs ex vivo*

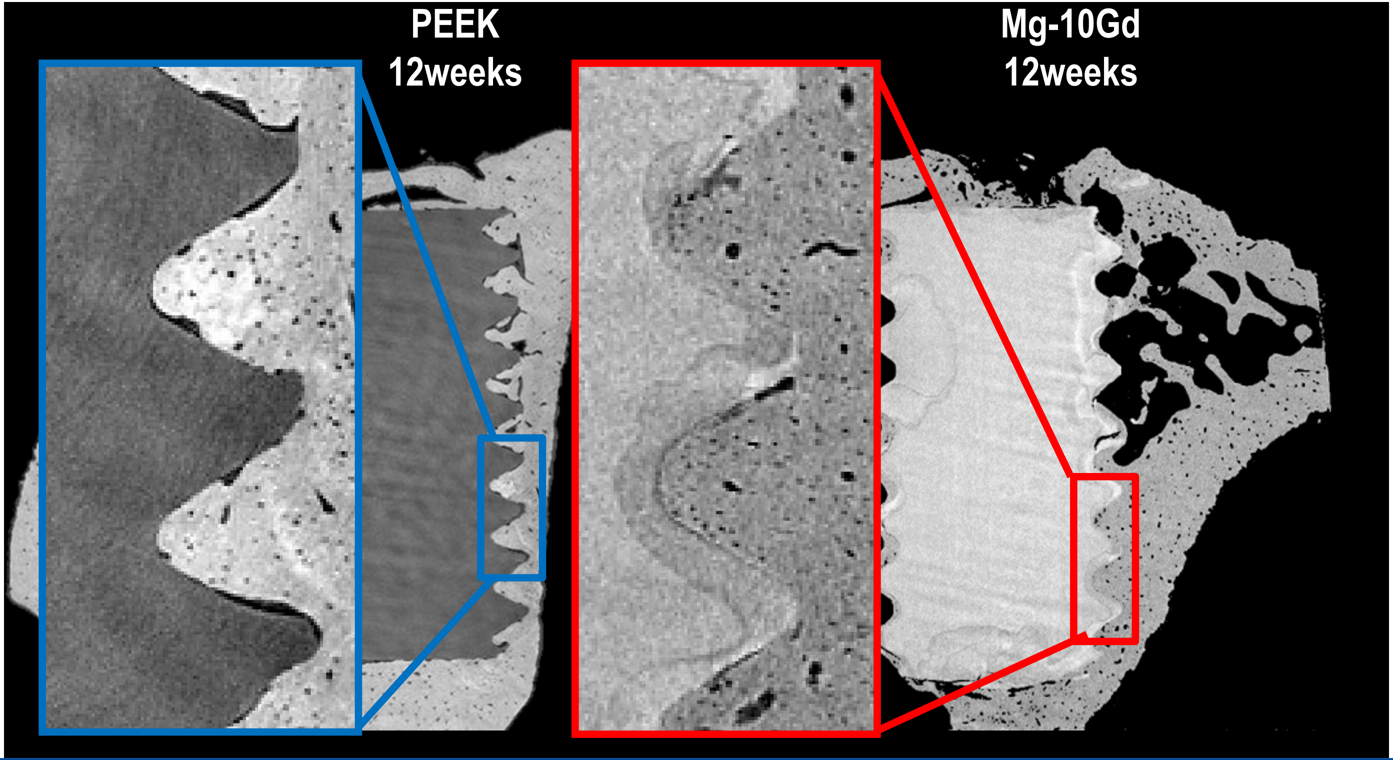




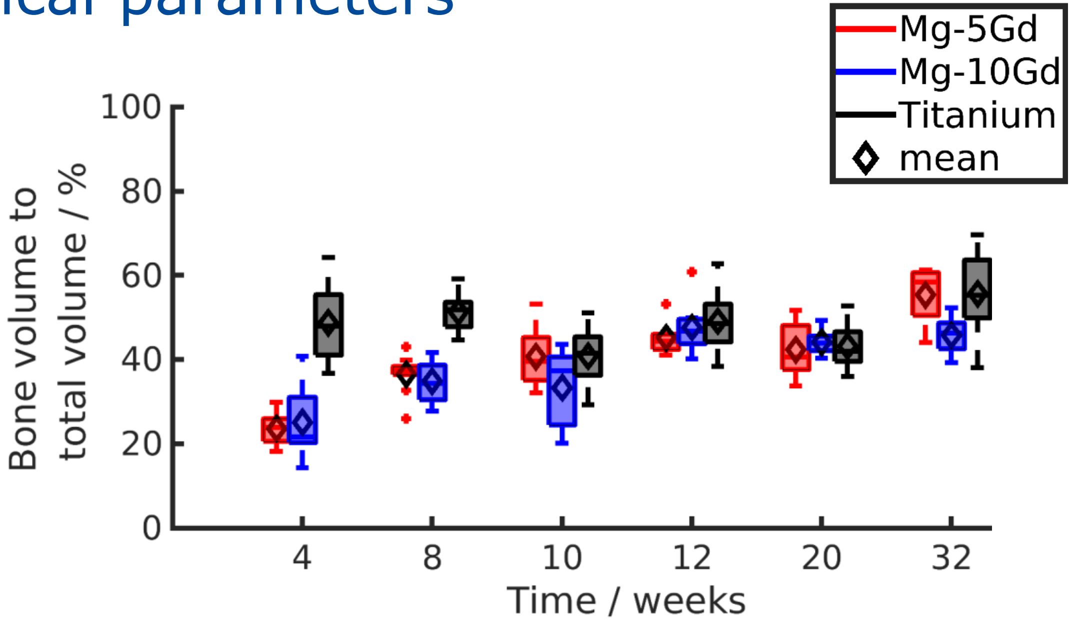
Diana Krüger *et al.*, Bioactive Materials 2021; Diana Krüger *et al.*, Magnesium and alloys 2021

Static SRµCT *ex vivo*

Diana Krüger *et al.*, Bioactive Materials 2021



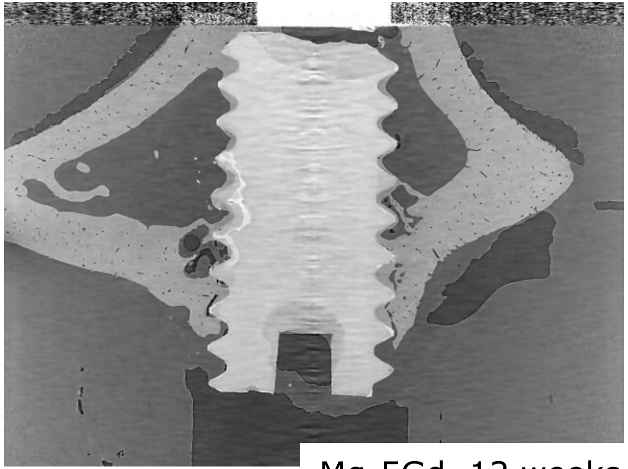
Morphological parameters



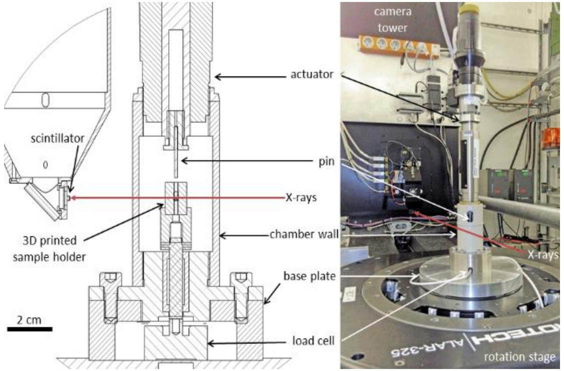
Diana Krüger *et al.*, Bioactive Materials 2021
Iskhakova and Cwieka and *et al.,* Bioactive Materials 2024

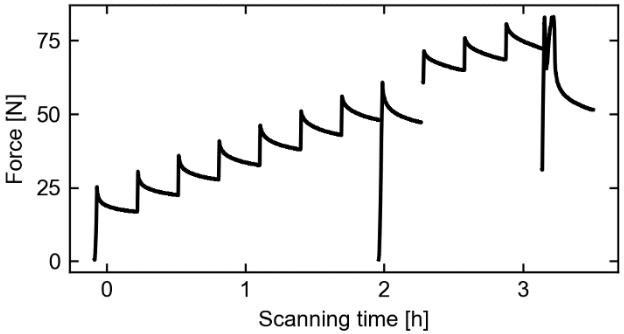
In situ loading experiments

- Testing of the bone-implant interface
- Step-wise loading
  → Force increment: 2.5 N, 9 steps



Mg-5Gd, 12 weeks

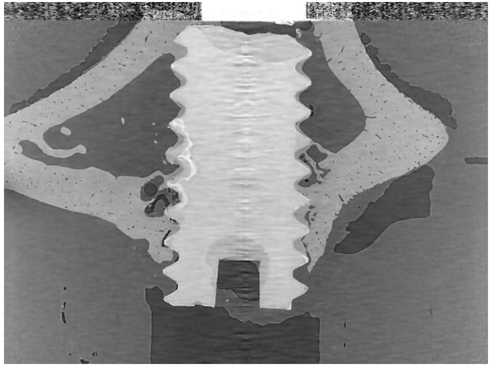


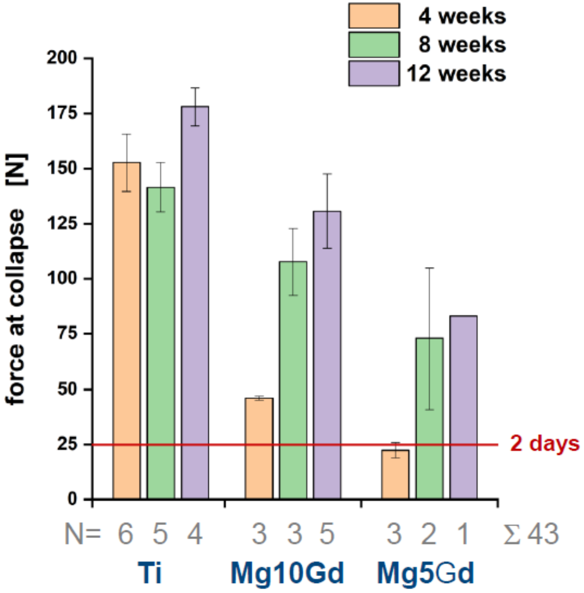


Courtesy of Stefan Bruns and Julian Moosmann, Hereon

In situ push-out experiments - Ultimate Loading Force

Stefan Bruns





- Depending on the degradation the integration into bone is changing
- Mg-xGd requires longer time for a good mechanical performance
- BIC or BV/TV aren't good predictors for all implant types

Bruns et al., Bioactive Materials (2023)