From Nano- via Micro- to Macroporosity:
Synchrotron Radiation Based Microtomography of 3D-Printed Ceramics

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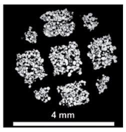
INTRODUCTION



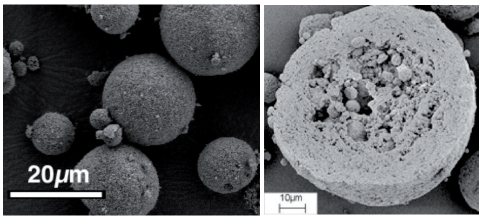
Rapid prototyping technology, especially 3D printing (3DP) has become attractive for fabrication of bone augmentation scaffolds. One main advantage of 3DP is the ability to create scaffolds with controlled open porosity for osteointegration [Gaulthier Biomater. 19 (1998) 133-139]. Porosity analysis on the different length scales, however, is a major challenge. Synchrotron-radiation-based micro computed tomography (SRµCT) at HASYLAB/DESY provides the micrometer resolution to extract the exact morphology down to 1µm. In addition, based on average values one can determine the total porosity of the nanometer scale. Consequently, SRµCT is a powerful method for porosity analysis.

MATERIALS AND METHODS

Spray-dried Hydroxyapatite (HA) granulates were used as building units for the 3D printing process. The highly porous granules show a multimodal size distribution with maxima at 25, 50 and 120 µm. Organic additives were supplied during the spray-drying process to enhance the flowability of the granules as well as the interaction of the material with the binder liquid during the 3D printing process.

3D printing technology allows the fabrication of real parts directly from 3D computer data. CAD as well as medical data (e.g. CT Scans) can be used for scaffold or implant design.

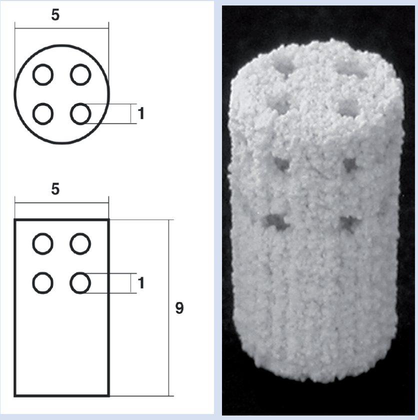
Prior to the fabrication step, the computer data have to be separated in several slice images. These slices can be processed in the 3D printer layer by layer [Seitz et al. Appl. Biomater. 74B (2005) 782].



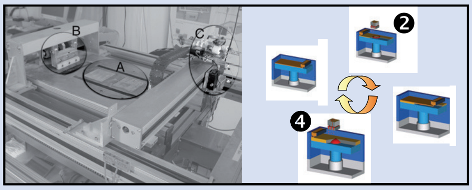
SEM- Images of HA-granules used for 3D printing. The left image shows a cross-section of a selected granule revealing the internal nanoscale porosity of the material.

The building process itself consists of four steps: First, a thin layer of granular material is deposited on the building platform. Second, one slice of the dataset is printed with a liquid binder onto granulate. Then, after the building platform is lowered, a new material layer is deposited and the printing process restarts until all slices of the dataset are printed. Finally the model can be removed from the building site. The HA-scaffolds for this study were fabricated using an experimental 3D printer with a spatial resolution of about 100dpi and a slice thickness of 200 µm. Subsequently, the scaffolds were sintered at 1250 °C to enhance their mechanical stability.

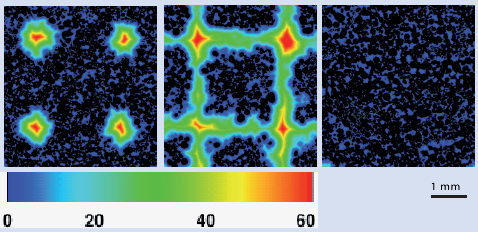
SRµCT was performed at the beamline BW 2 (HASYLAB at DESY, Hamburg, Germany) operated by GKSS in absorption contrast mode using the photon energy of 24 keV and the voxel size of 4.1 µm. 3D data were reconstructed by the filtered back-projection algorithm.



Design of the 3D printed scaffolds used in this study. The right image shows the 3D printed scaffold after sintering.

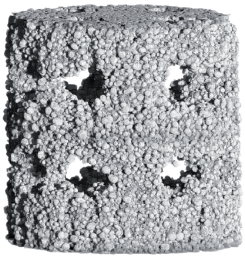


3D printer comprising of building platform (A), material recoater (B) and printhead (C). The right image shows a schematic process workflow of 3D printing.

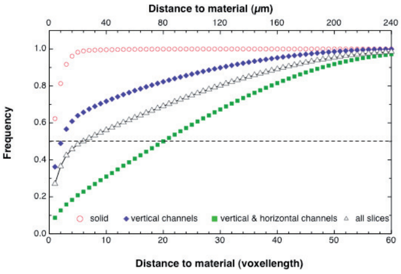


Distance transform maps of three selected region of interest (ROI) of the 3D printed HA-scaffold. The colors represent the distance of the voxel to material.

POROSITY ANALYSIS



3D reconstruction of SRµCT dataset of a 3D printed HA-scaffold



Porosity (distance to material) of 3D printed HA-scaffolds. The open triangles represent the trend of the whole scaffold whle the other curves show information of the three ROIs.

Due to the porous structure of the granules, a threshold determination was difficult. Therefore the dataset was divided into three parts: without channels, with vertical channels and including both, vertical and horizontal channels. For binarisation of the data, a threshold of 111 was selected for further calculations [Irsen et al. Proc. SPIE 6318 (2006) 631809].

The 3D-distance map allows the quantification of the pore structure of the scaffold. Here the minimal distance of each voxel to material is determined. The overall mean distance is 24 µm. Because the scaffold structure is inhomogeneous, three different areas of the dataset were analyzed. Here the mean distances vary from below 4 µm for the solid material to 82 µm for the region with vertical and horizontal channels.

CONCLUSION AND ACKNOWLEDGEMENT

The true micrometer resolution of SRµCT allows the precise determination of the granules shape, the pore sizes and the pore size distribution of the investigated porous scaffolds. The interconnectivity of 3D printed pore structures can be analyzed on the scale well below the cellular level. Furthermore, the determination of the integral nanoporosity becomes possible non-destructively. This information is crucial for the optimization of the scaffold fabrication process and, finally, for the osteointegration.

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