Nondestructive 3D insights into foamy metals and bioregenerative ceramics

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Micro-structured, multi-component material systems are of high interest due to their broad range of application fields. Within this article we focus on two representatives of these material systems and the insights we can obtain by employing X-ray microtomography.

Aluminum foams, with their high specific stiffness are of substantial interest for lightweight constructions e.g. for car bodies. But on the way to industrial applications there is still a need for basic approaches to control their final pore structure [1].

Something quite different are biodegradable ceramics (Fig. 1). These compounds can be used alternatively to autogenous bone grafts for supporting the bone regeneration in defects, which can occur after tooth loss for instance. Different biocompatible materials are available on the market. But there is still a demand for detailed investigations in order to optimise their properties for the various clinical applications [2].

In order to follow the pore formation in early stages of metallic foams and the biodegradatation of bone substitute materials, we employed microtomography (µCT) using X-rays. This method is well established to image the inner structure of a sample in three dimensions in a nondestructive manner. µCT is particularly powerful when it is used in conjuction with synchrotron light sources like BESSY, since these sources offer a photon beam with a nearly parallel propagation, a high flux density, and a partial spatial coherence. The advantages are obvious: Resolutions up to sub-micrometer, different contrast modes (e.g. absorption contrast, holotomography, refraction contrast) and high data acquisition speed [3, 4, 5]. Furthermore, the high quality of volume images acquired with synchrotron-based µCT allows to perform a subsequent 3D image analysis [5, 6].

We used the excellent microtomography facility of the BAMline for our experiments [7] using monochromatic synchrotron radiation for our microtomography scans. This enabled us to distinguish between different phases of materials in the resulting volume images due to their different attenuation of the X-rays: e.g. the matrix and the blowing agent's particles in metallic foams or ceramic particles and bony tissue (Fig. 1, 2). In the subsequent 3D image analysis we separated different material phases within the images into Boolean images. These contain only the morphological information, marked with a Boolean '1' while the background is set to '0' [6].

By dilating the pore structure in successive steps we can determine the dependence of the blowing agent's (TiH_2) density on its distance to the pores space. Therefore, we are able to invest spatial correlations between the TiH_2 particles and the pores [8]. For aluminum foams produced from a pre-alloyed powder Al6061 (aluminum, silicon) it is known that pores and blowing agent's particles are spatially correlated in all foaming stages [9]. In AlSi6Cu10 and AlSi6Cu4 produced from a mixture of pure aluminum, copper and silicon powders we could prove spatial non-correlations between the TiH_2 particles and the early pores. This surprising result points out that there are different pore creation modes in the early stages of metallic foams which influence the spatial position of the first pores in the evolving foam matrix.

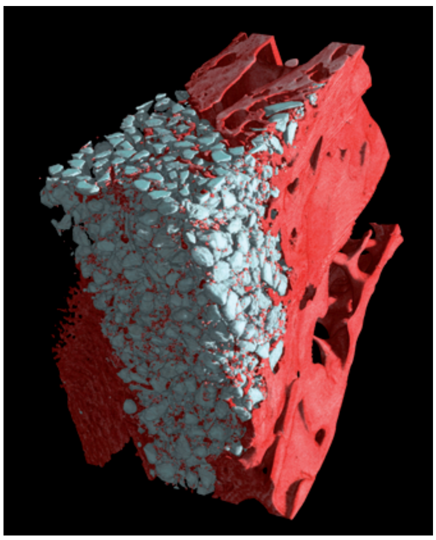

Fig. 1:
Bioceramic particles (white) three months after implantation in sheep bone (red) [8].

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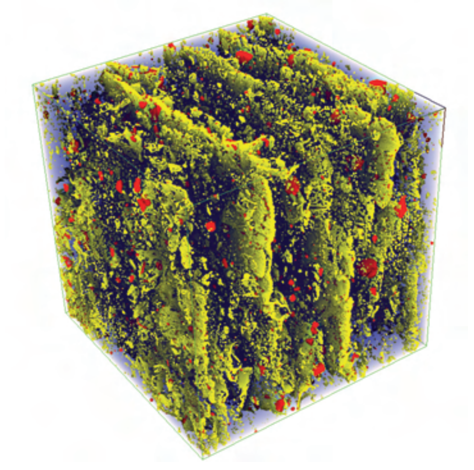

Fig. 2:
AlSi7 foam in early stage (6% porosity) with foam matrix (semitransparent blue), pores (yellow) and blowing agent (red).

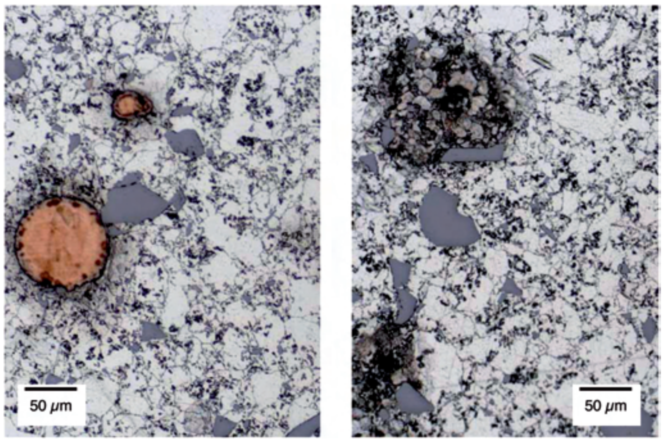

Fig. 3:
Metallographical photo of raw precursor AlSi6Cu4 (left) with Cu particles (brewn), Al (light grey), Si (blue) and tempered sample (right, same color code) [8].

The control mechanism behind this different behavior can be understood by examining etched and polished slices of the investigated samples (Fig. 3): the raw precursor contains pure copper, aluminum and silicon, the tempered sample only aluminum and silicon. Copper and part of the aluminum form an eutectic alloy already at lower temperatures. Here, gas set free from TiH_2 can nucleate most easy and form the first pores. In opposite to that precursors made of Al6061 are homogeneous due to the use of pre-alloyed aluminum with silicon. There are no 'weak' positions where a preferred pore creation can happen.

By masking the noise-reduced Boolean images of the bony tissue and the ceramics volume in the 3D image analysis, we obtained images which contain the morphology as well as density information of the different phases of material. Combined volume rendering of these data sets delivers excellent images which can be used for qualitative comparison with histological photos (Fig. 1). Samples were prepared as part of an animal study for comparing different bioceramics. We found a large difference in the amount of detected bone between the histological and the tomographical investigations (Fig. 4). The newly formed bone, if not fully developed, has a lower density and therefore is not visible in the µCT scans due to its low X-ray attenuation, while histology can detect it there due to the chemical sensitivity. This allows for a quality control of the bone regeneration depending on the bioceramic material used. The ceramic implanted in the specimen shown in Fig. 2 is degrading slowly: three months after implantation almost all ceramic particles are still left, only a small amount of bone tissue has formed. Bioactive ceramic materials with a higher biodegradability, which were also investigated in the context of this study resulted in an almost fully evolved bone structure after the same amount of time.

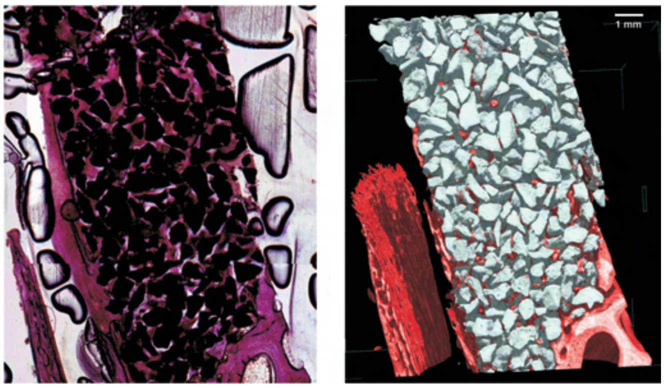

Fig. 4:
Left: histological image of bone substitute particles (black) in sheep sinus (bone in pink) and the rendered corresponding section from the segmented 3D data set (right) [8].

Our investigations show that heterogeneous precursors are one approach to control the pore structure of metallic foams as first pores start to grow on weak material positions. For the bioceramics we developed a novel approach to determine the stage of development of newly formed bony tissue by comparing histological with (synchrotron-)tomographical slices.

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