Nano-scale 3D reconstruction of C-S-H-phases using modern FIB/SEM technology

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Introduction

Modern Scanning Electron Microscopes (SEM) allow high-resolution imaging of the nano- to microstructure of building materials like cement and concrete. Until now, the structural exploration has mainly been carried out on fractured or polished 2D surfaces. While this approach was able to unveil many details like the shape of C-S-H-phases, it was not possible to create a digital 3D reproduction of those shapes.

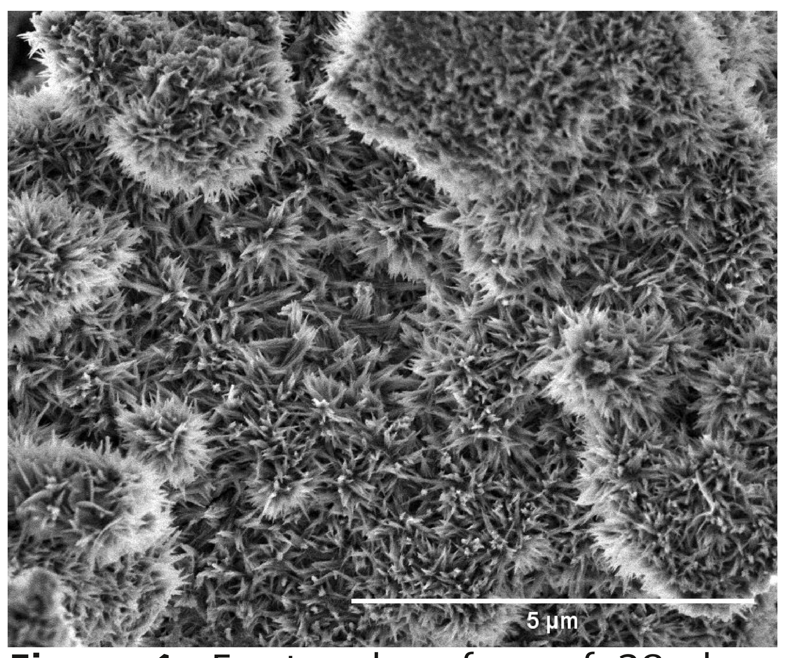

Figure 1: Fractured surface of 28 days hydrated alite overgrown with needle shaped C-S-H phases. (SE, 2 kV)

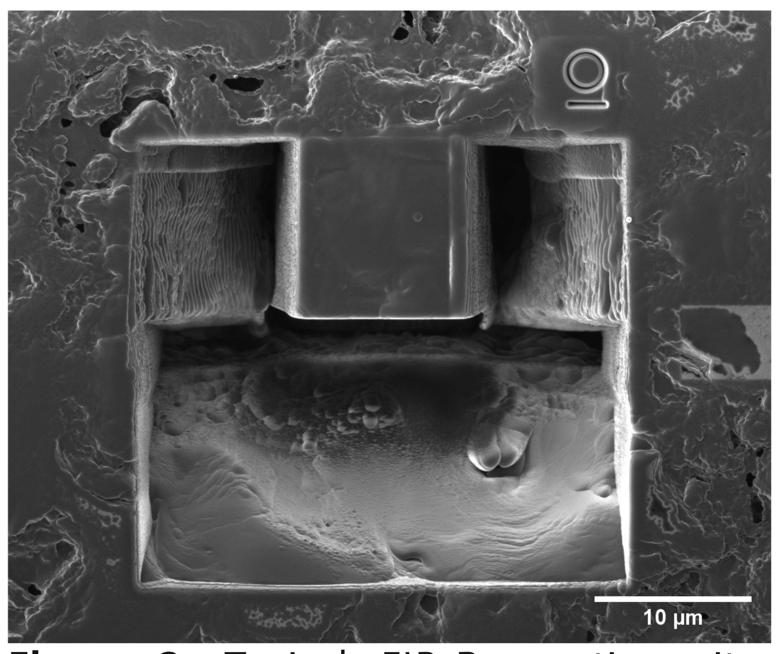

Figure 2: Typical FIB-Preparation site. The cube is 10 x 10 µm and will be cut to 10 nm slices. (Ion-Image, 30 kV)

Experimental challenges

The Focused Ion Beam (FIB) technology is already widely adopted in the semiconductor industry, cell biology and to analyse the pore structure of battery materials, oil shales and clay material [1, 2]. It utilizes a gallium ion beam to cut layer by layer of a material volume and then using the various SEM detectors to image and analyse the revealed surface. This allows to reconstruct a detailed 3D-volume of many materials. While some approaches to image several cubic micrometer small volumes of hydrated alite already exist [3], none of these approaches were able to deliver a detailed 3D-volume of the C-S-H-needle structure (Figure 1) due to the sensitivity of the material to the electron and ion beam. Using low voltages for the electron beam is key to obtain undisturbed images. Furthermore, for those relatively porous specimens, a resin intrusion is essential to avoid artifacts caused by the pore background.

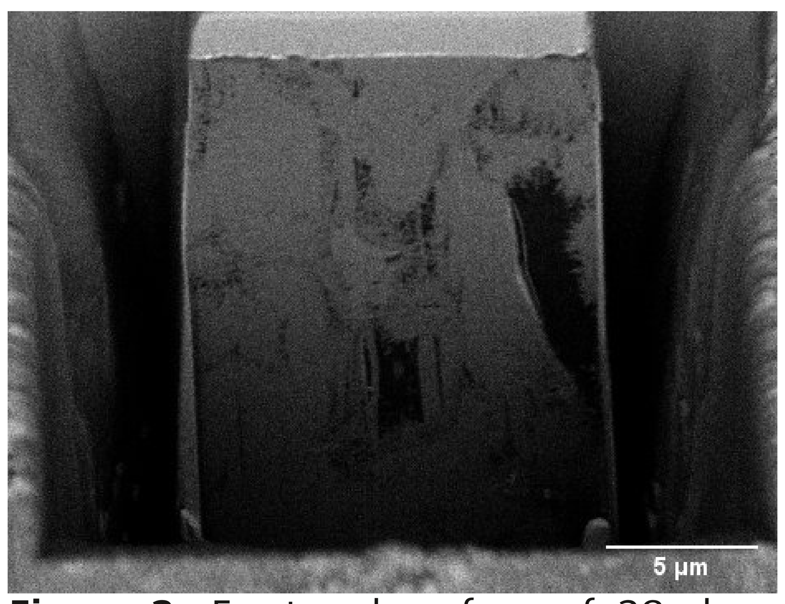

Figure 3: Fractured surface of 28 days hydrated alite overgrown with needle shaped C-S-H phases.

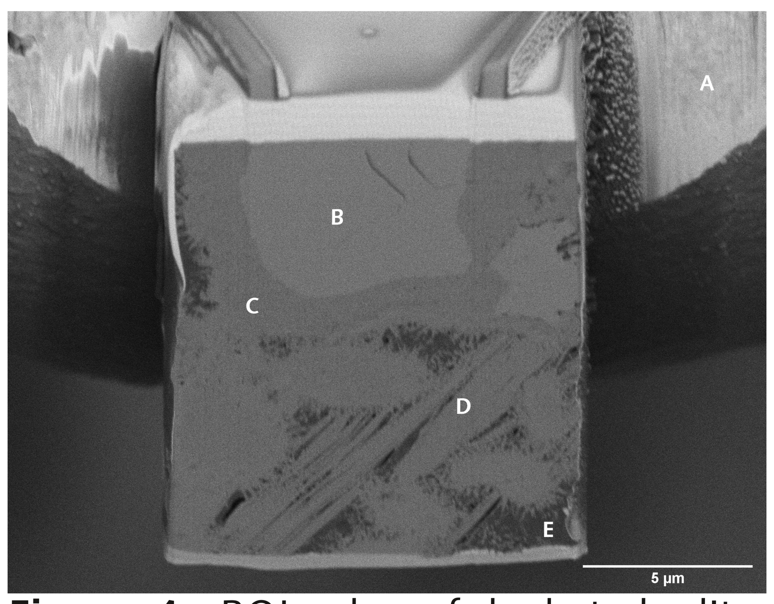

Figure 4: ROI-cube of hydrated alite, welded to a copper grid (A). (B) alite, (C) C-S-H, (D) CH, (E) resin. (SE, 2 kV)

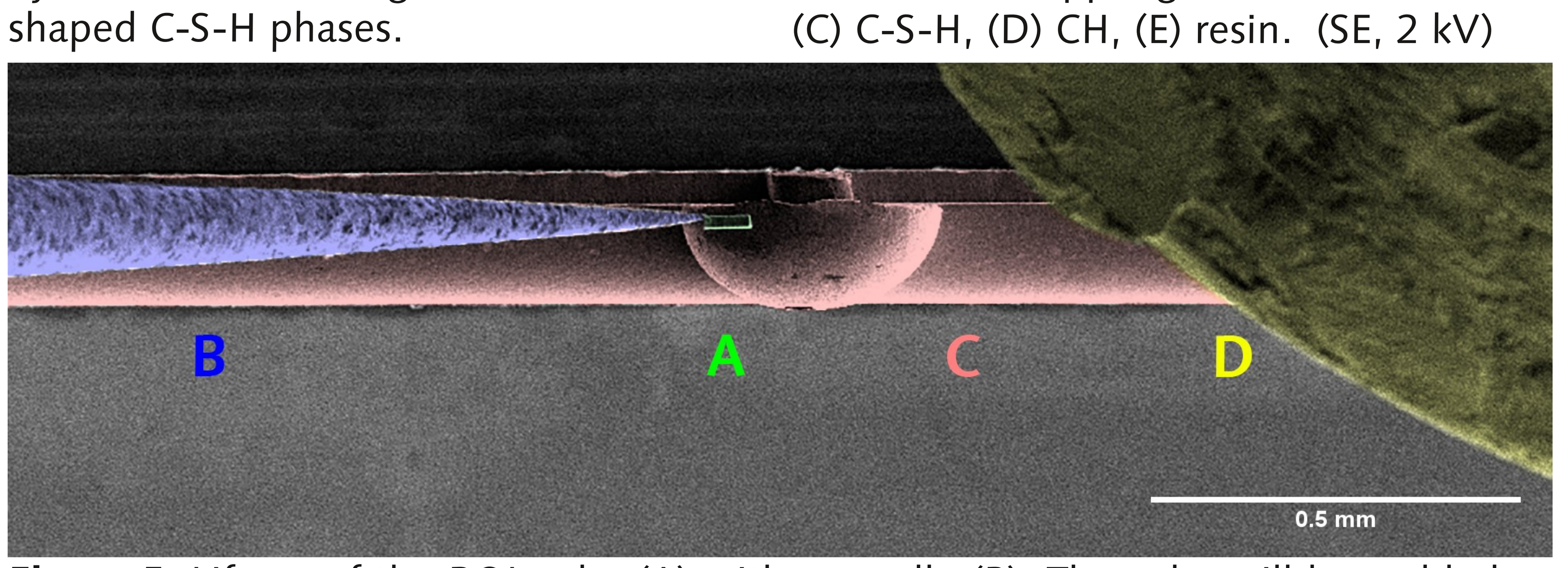

Figure 5: Liftout of the ROI-cube (A) with a needle (B). The cube will be welded to a copper grid (C) using a platin deposition system (D), (SE, 2 kV)

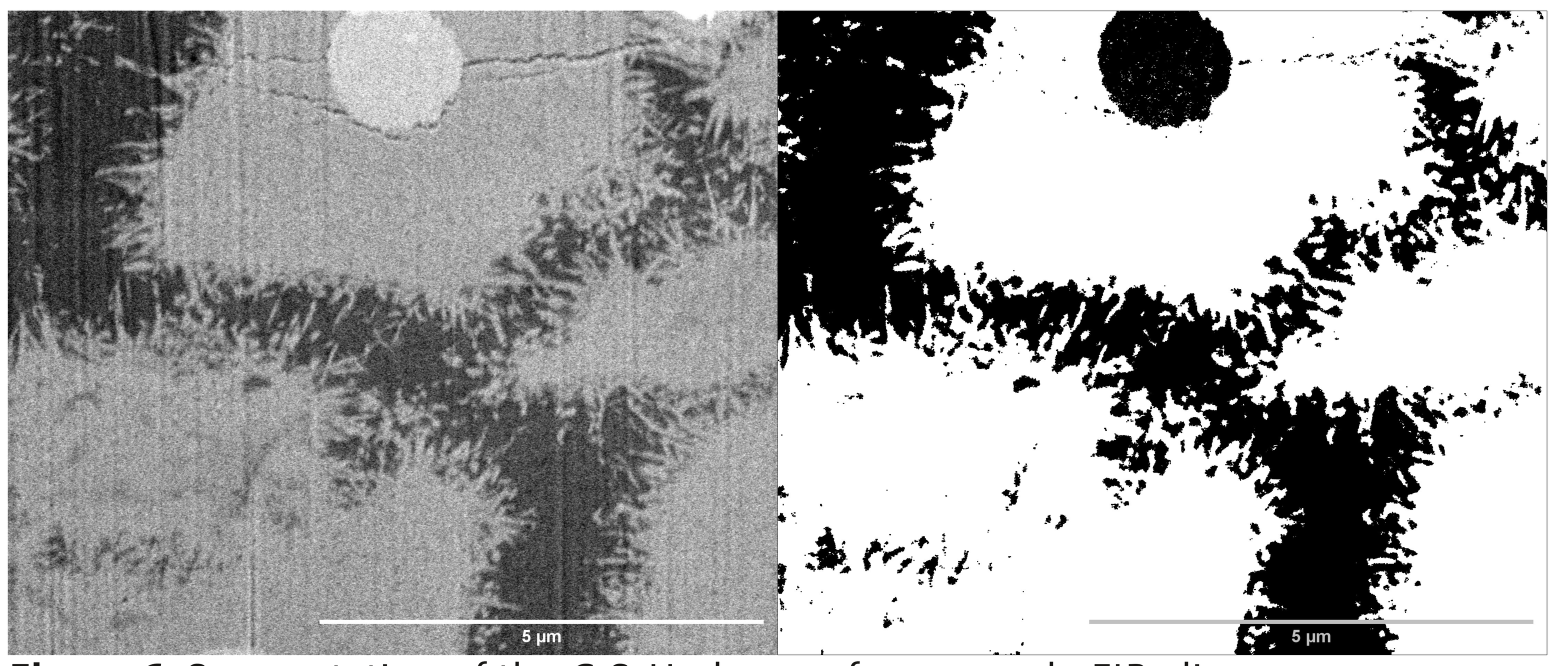

Figure 6: Segmentation of the C-S-H phases of an example FIB-slice.

The preparation site for a typical FIB process is shown in Figure 2. The electron beam scannes the surface to be evaluated at an angle of 52°. Since the region of interest (ROI) remains connected to the bulk material, a shadowing effect can be observerd (Figure 3). While this effect may be corrected in some degree by image manipulation, the degrading signal quality bevcomes an issue using less sesitive detectors like EDS-detectors. Furthermore, charging artefacts become an issue in the progession of the imaging process, since the material is non-conductive. Therefore, the ROI-cube will be lifted out of the bulk material as visualized in Figure 5. This leads to an improved image quality (Figure 4).

The resulting image stack is easyly segmentable as demonstrated in Figure 6 and can be reconstructed as a voxel based or an triangulated model. This 3D-volume can be further processed so that quantification of solids and pores at nanoscale dimension is possible.

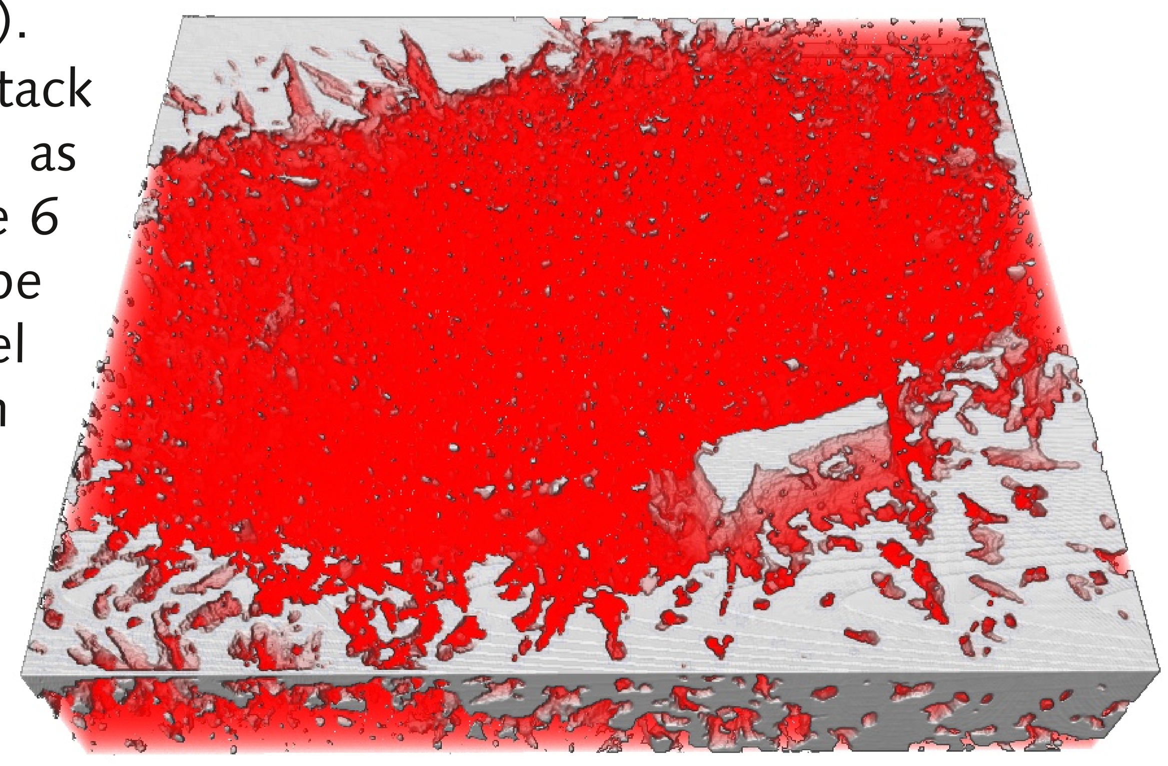

Figure 7: 3D-reconstruction of pores (solid, grey) within dense inner C-S-H (semi-transparent, red) of a 28d hydrated alite specimen (3.7 x 3.3 x 0.74 µm³).

Results

The 2D images (Figures 4 and 6) and the respective reconstructions (Figures 7 to 9) show, that it is possible to obtain a close-to-native 3D-representation of the nano-structure of hydrated alite pastes. It was possible to obtain a voxel resolution down to 4 x 4 x 10 nm as shown in Figure 1. Results of 3D data analysis will be compared with conventional 2D analysis. The obtained pore connectivity in 2D and 3D analysis will we discussed.

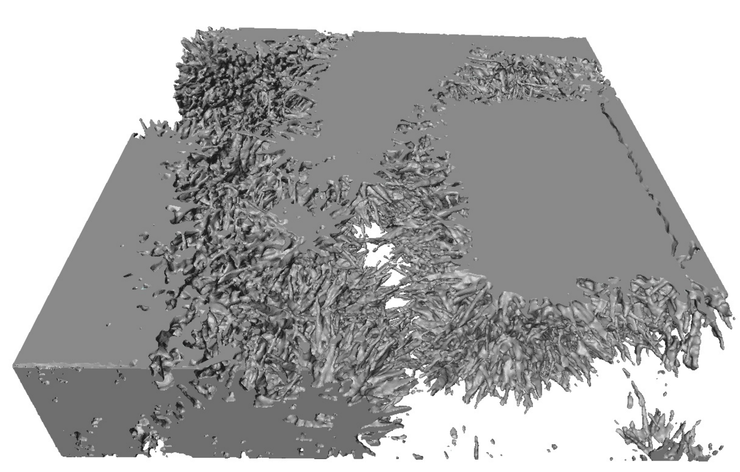

Figure 8: Voxel based 3D-reconstructions of the needle structures of C-S-H phases in hydrated alite.

Figure 9: Triangulated surface of the needle structures of C-S-H phases in hydrated alite.

References

[1] J. Goral, I. Walton. M. Andrew, M. Deo, Fuel 2019, 258 (DOI: 10.1016/j.fuel.2019.116049)
[2] S. Gaboreau, J.-C. Robinet, D. Prêt, Microporous and Mesoporous Materials 2016, 224 (DOI: 10.1016/j.micromeso.2015.11.035)
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