P11: Characterisation μ-cracks in concrete and transport in concrete

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1. Introduction

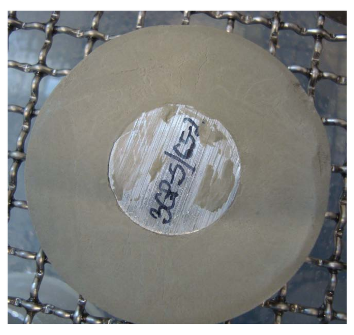
Durability of concrete can be described using the concept of service life: “the time during which a concrete fulfills its performance requirements”, without non-intended maintenance. Durability depends not only on the original material composition and properties, but also on the environmental actions during service. Understanding transport processes and knowledge of the microstructure of cementitious systems will allow to predict the durability of cementitious materials and reduce energy consumption in concrete manufacturing. Two elements of microstructure of cement-based materials, porosity and cracks, play a key role in transport processes. New experimental techniques for the characterisation of porosity and micro-cracks in mortar and concrete in 3D can provide important informations about the effect of microstructure on mass transport, which is a critical aspect for predicting durability of concrete.

3D-microscopy methods, including Focused Ion Beam nano-tomography (FIB-nt) and X-Ray Tomography (micro-CT) are used to characterise nano- and micro-cracks in cement based materials. FIB provides a nanometric resolution of the three-dimensional distribution of cracks and porosity, thus enabling a tomographic reconstruction of samples ranging from a few hundreds of nanometers to a few microns in thickness. On the other hand, the micro-CT approach extends the scale of study up to few tens of microns. The application of these techniques to the study of cementitious materials has been quite limited until now. In this work, we report on preliminary results that show a feasibility of these techniques to map and quantify the parameters that will eventually play an important role in the modelling of water transport and, by extension, the prediction of the durability on the basis of the multiscale identification of micro-cracks, micro and nanoporosity, reaction products and detailed distribution of cement components.

2. Samples preparation

Cement paste

w/b	0,4
binders	OPC, OPC+Slag (70%), OPC+Silica Fume (10%)
samples shape:	disc Ø 65mm, 10mm height, casted with steel and mortar rings (Ø30mm, 10 mm high)
curing time	7 days
drying condition	RH=50%, T=23°C, one direction drying

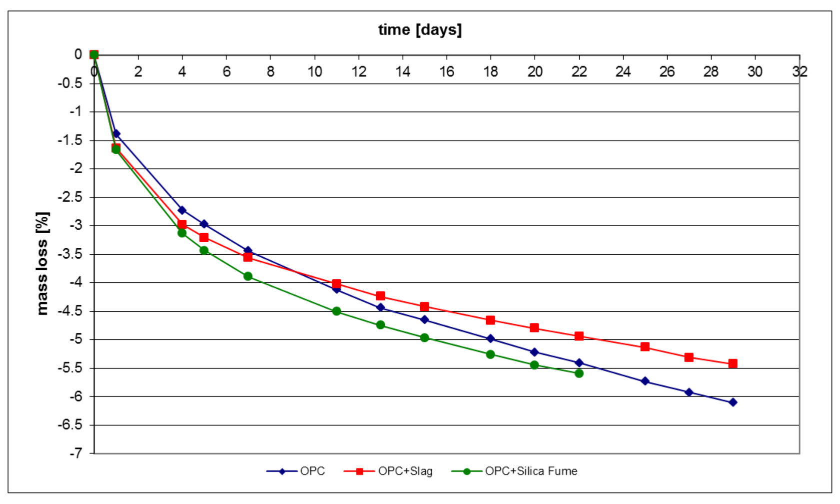


All samples were produced at Sika in Zurich, for future investigation they will need more preparation steps:

X-Ray CT
Cutting for 10x10x10mm³ and 5x5x5mm³

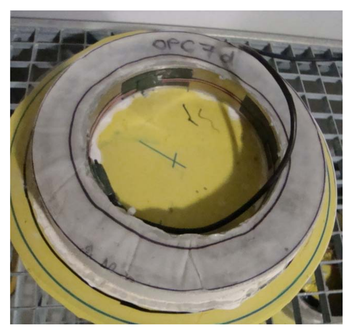
FIB-nt
Vacuum impregnation of epoxy resin
Grinding and polishing
Carbon coating
Fixing on sample holder

Moisture loss:

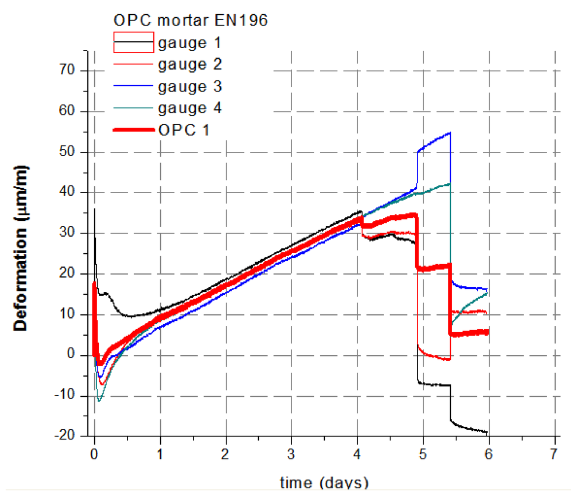


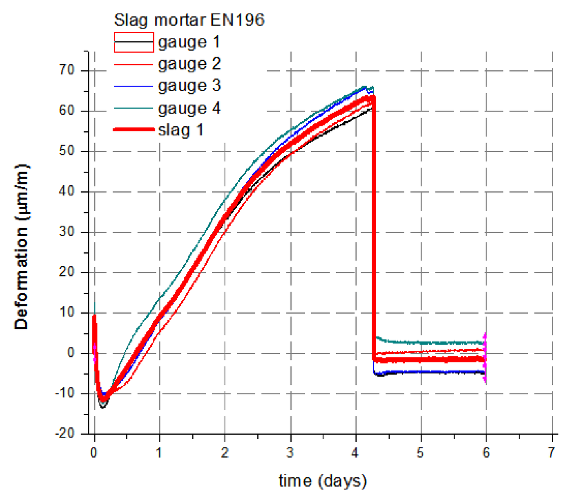
Mortar

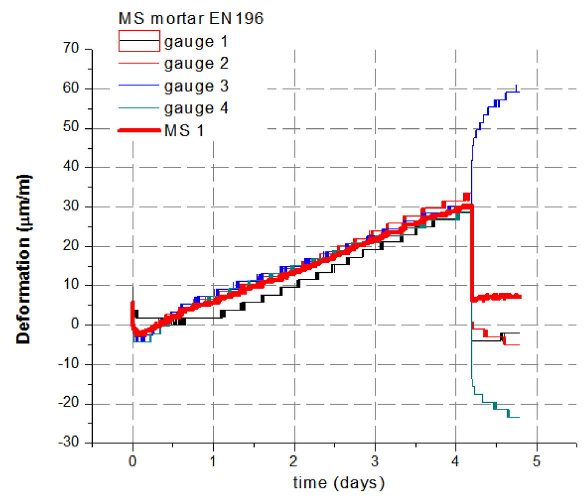
w/b	0,5
binders	OPC, OPC+Slag (70%), OPC+Silica Fume (10%)
samples shape:	ring Ø 200mm, 40mm height, 40mm wall thickness, casted with steel ring – standard ring test
curing time	7 days
drying condition	RH=50%, T=23°C, one direction drying



Ring test results:

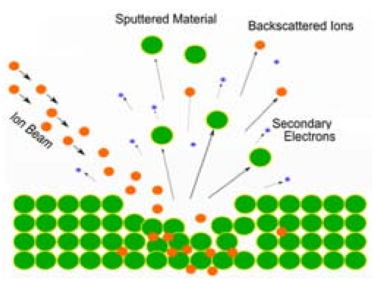


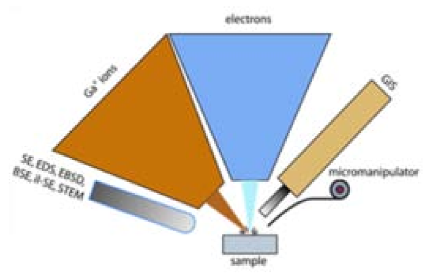




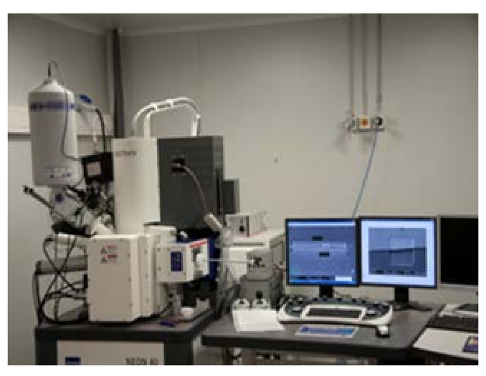
3. Focused Ion Beam nano-tomography

Focused Ion Beam system produces and directs a stream of high-energy ionized atoms (liquid metal ion source - LMIS) of a relatively massive element (Ga⁺), focusing them by an electric field onto the sample for etching or milling the surface and as a method of imaging. The ions easily expel surface atoms from their positions and produces secondary electrons from the surface.

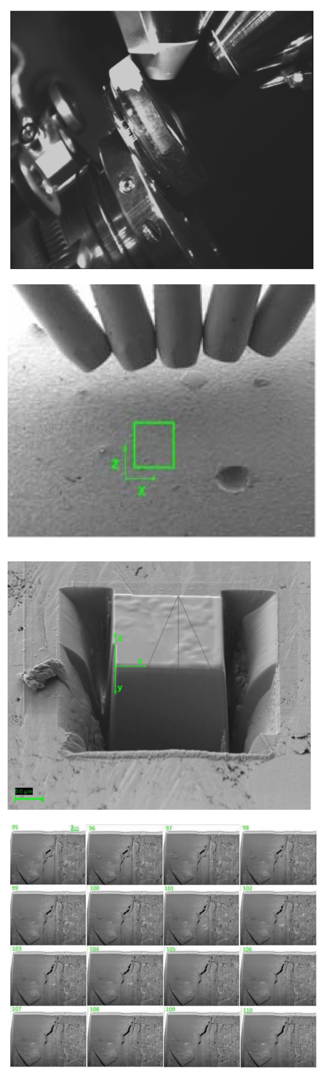




FIB nano-tomography is a process in which the sample is sectioned with Ga⁺ ions and the image is obtained with the SEM in the Dual Beam SEM-FIB System. When all the individual images are aligned, the 3D reconstruction of the structure is obtained.



Specimen cube prepared for serial sectioning procedure



The sample is placed on a stage in the microscope chamber. The stage is tilted at 54º so the sample surface (x-z plane) is perpendicular to the ion beam (y-direction).

The optimal focusing, contrast, brightness and magnification are adjusted. Then the interested region is chosen and a very thin Pt layer is deposited. The metal coating reduces charging effects and protects the surface from undesired ion-induced erosion.

In order to render the x-y plane accessible for image acquisition a trench is eroded in front of and at both sides of the cube. This evades the shadow effects from the side wall. Additionally reference marks are placed on the top of the cube for the drift correction in x-y planes.

When a sample is prepared the serial sectioning procedure is started.First a thin layer of material from the x-y plane is eroded with the ion beam and the picture ofnewly exposed surface is acquired by SEM. In this way a stack with hundreds of images is produced while moving through the sample in the z-direction.

Data processing and reconstruction of 3D structures

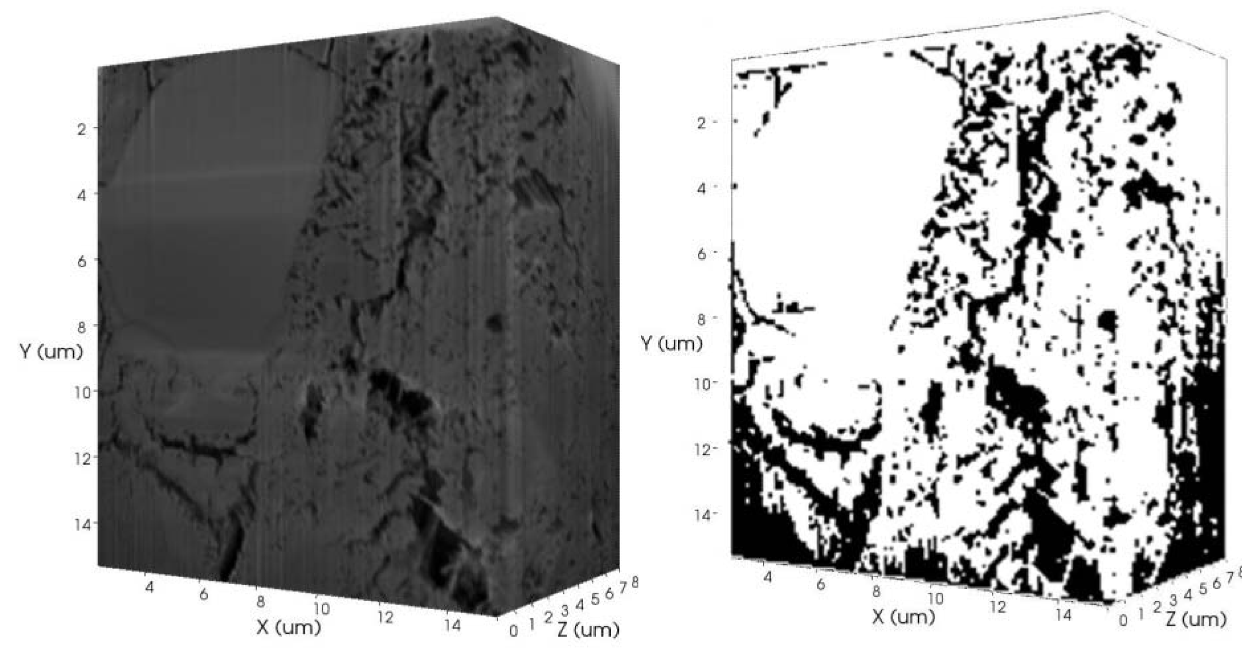
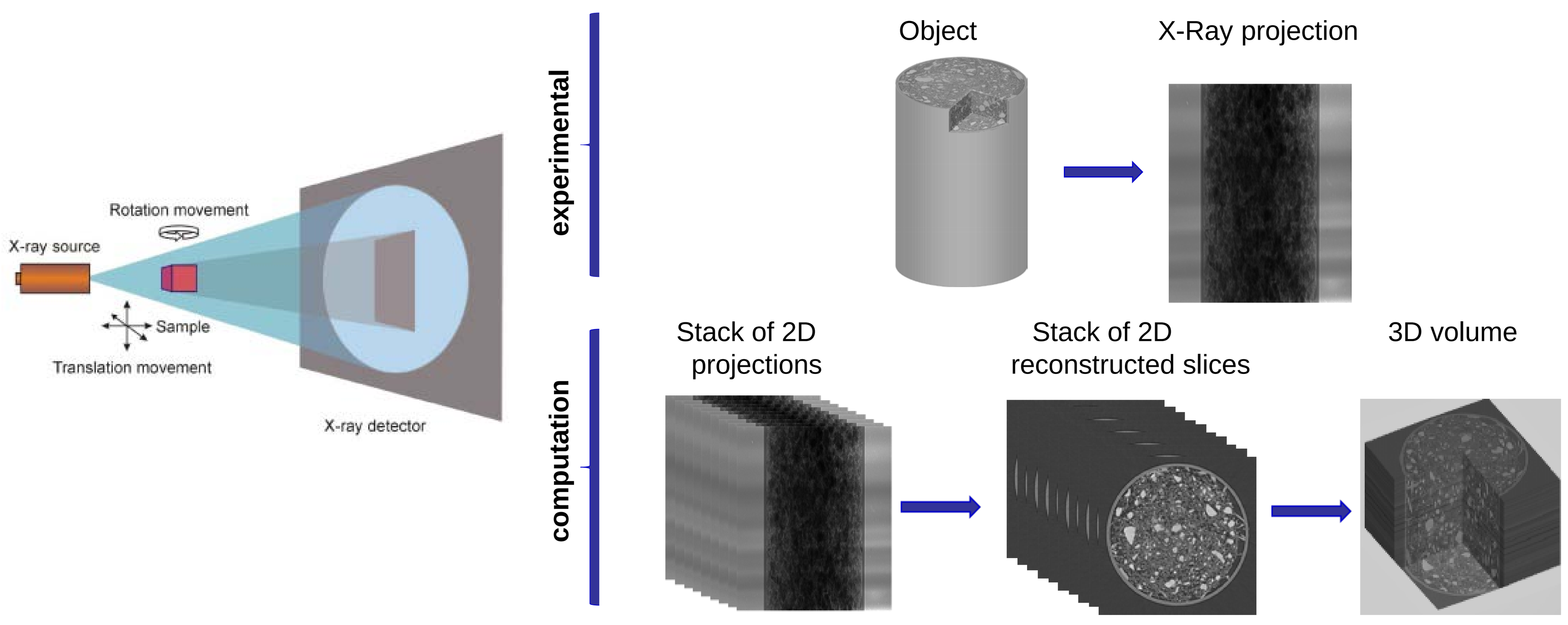


Image processing is made in 3D reconstruction software – Fiji and includes the following steps:
1) correction of voxel dimensions
2) alignment of image stack and overlay correction for x-y drift
2) crop-out of a suitable subvolume
3) segmentation and binarization
4) parametrization and measurements.

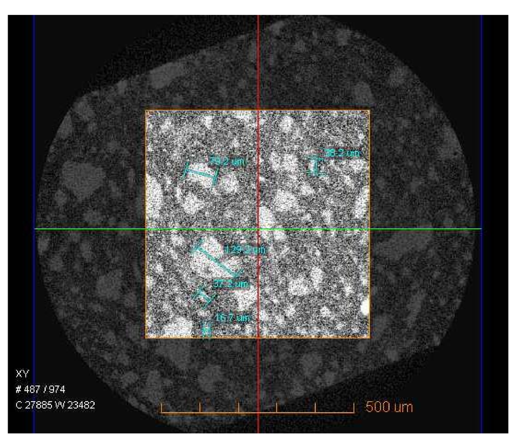
4. X-Ray Computed Tomography

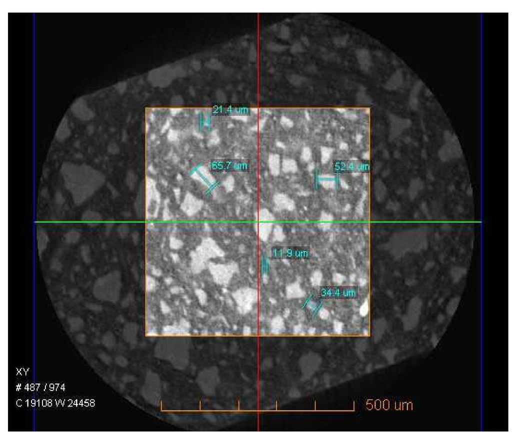
X-Ray Computed Tomography (CT) is a nondestructive technique for visualizing interior features within solid objects, and for obtaining digital information on their 3-D geometries and properties. In this technique hundreds of 2D projection radiographs are taken of a specimen at many different angles. This series of radiographs is mathematically reconstructed to produce a quantitative 3D map of the object.

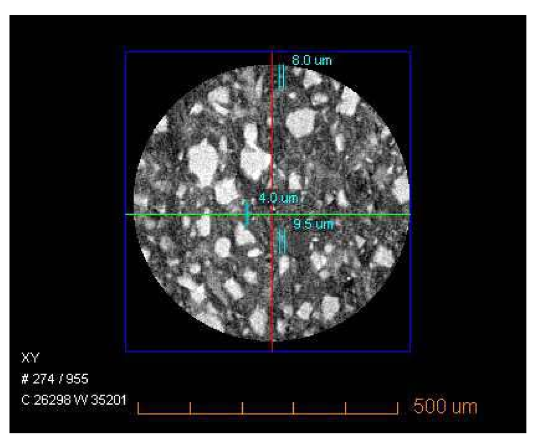


Tomographic scans were performed at the Slovenian National Building and Civil Engineering Institute in Ljubljana (ZAG) on the Xradia 400:

Sample size:	1mm²	**Sample size:**	1mm²	**Sample size:**	1mm²
Exposure time:	1 s	**Exposure time:**	15 s	**Exposure time:**	60 s
No of images:	4000	**No of images:**	4000	**No of images:**	4000
Pixel size:	1.190 μm	**Pixel size:**	1.190 μm	**Pixel size:**	0.598 μm
Voltage / Power:	80 kV / 8W	**Voltage / Power:**	60 kV / 8W	**Voltage / Power:**	60 kV / 10W
Objective lens:	20X	**Objective lens:**	20X	**Objective lens:**	40X







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