Experiment title:	Experiment number:
Tomographic analysis of micro-crack formation and growth in hardening high-performance concrete.	ME-214

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Report:
The aim of this experiment was to determine the appearance of microcracks in the cement matrix of high-performance concrete. Phase contrast emphasises interfaces between pores or cracks and the cement matrix.
Therefore, samples with different cement types and water-to-cement ratios w/c were examined during solidification by high-resolution computerized tomography in real-time. The non-hardened cement paste proved difficult to be investigated owing to its mechanical instability.
Nevertheless we could obtain 3D images of cement during hardening showing the evolution of porosity at the micron scale. In Fig. 1 an example of a slice reconstructed by CT is shown. It is possible to distinguish four characteristic linear absorption coefficients:

1. highly absorbing cement particles with a subtexture being inert during hardening (very light),
2. non-absorbing pores which are emphasized by phase contrast at their border and whose volume fraction grows with proceeding hardening time due to autogeneous shrinkage (black),
3. the initial, weakly absorbing cement stone solidified from the cement paste (dark gray) and
4. the final hydration product, the highlier absorbing cement stone (light gray).

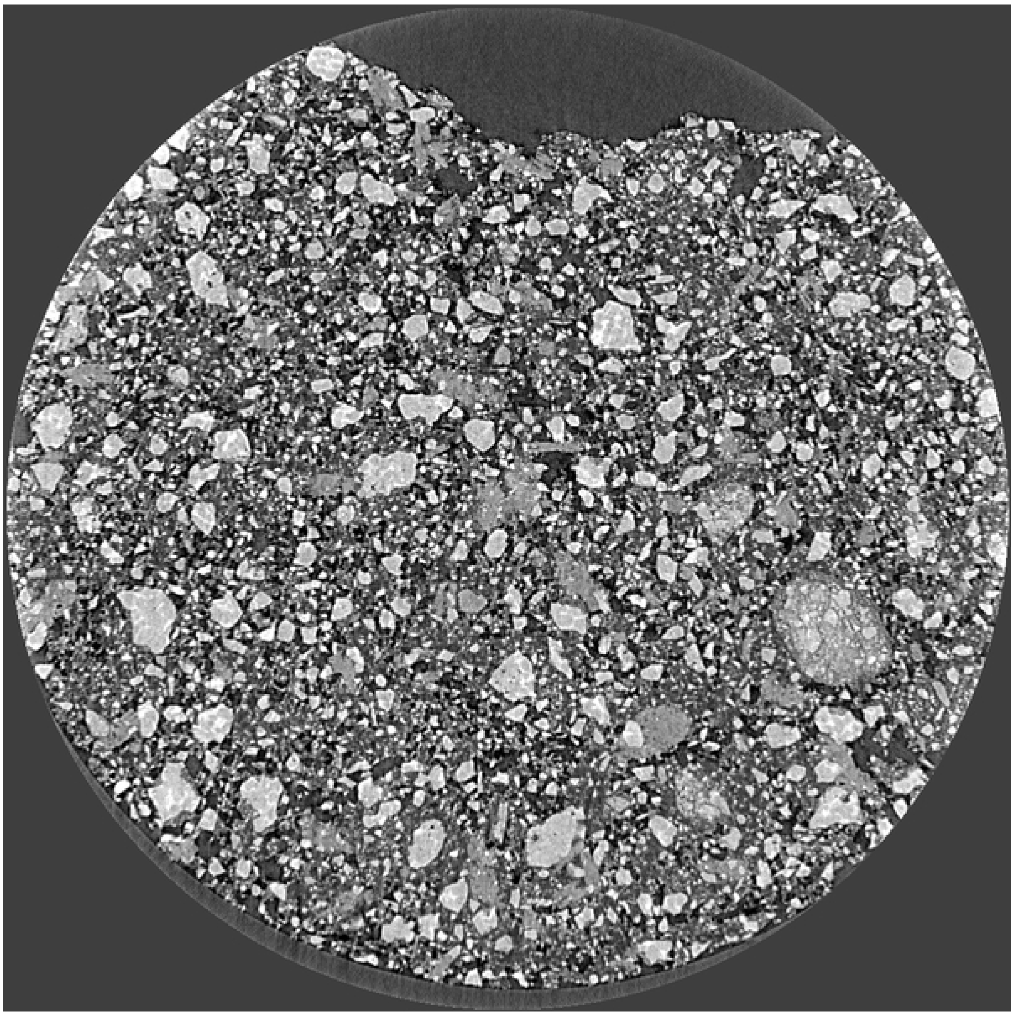

Figure 1: Tomographic slice through a cement sample after 16.5 hours of hardening. On the bottom half there is a crack / pore band extending between the left and right sample vessel wall. Image size is 1024×1024 pixel with a size of 1 μm.

Pores are sometimes aligned with respect to other pores to form interconnected pore bands which stretch out between the walls of the cylindrical sample vessel.

During the experiment we explored the unexpected perspective to visualise the hydration of the cement (see Fig. 4). The chemical reactions yield differences in the local density distribution within the sample, visible both in the slices of the reconstructed 3D images as well as in their histograms The histograms in Fig. 2 show the distribution of the reconstructed linear attenuation coefficients μ on a region of $512 \times 512 \times 1024$ voxels. In conjunction with Fig. 4 we see that the cement grains remain essentially inert on the investigated time scale and the cement paste transforms from the unhydrated state towards a hydrated one which is more absorbing. Pores do develop during this process due to autogeneous shrinkage.

It is possible to quantify the porosity as a function of the time. From Fig. 3 we see that the porosity is increasing with time and that the capillary pore system gets more and more interconnected. After about 17 hours of hardening samples with a low cement content the hydration process proceeds asymptotically slow.

Furthermore, pore size distribution functions have been obtained directly from 3D data sets from a single sample during hardening.

These results show that synchrotron radiation allows to get new insight into the hydration process of high-preformance concrete. A future aim will be to investigate the influence of mineral and chemical admixtures on the hydration of high-preformance concrete in dependence of the water-to-cement ratio w/c.

The experience gained during this experiment with the solidification behaviour will presumably allow to investigate a larger number of samples within a reasonable time.

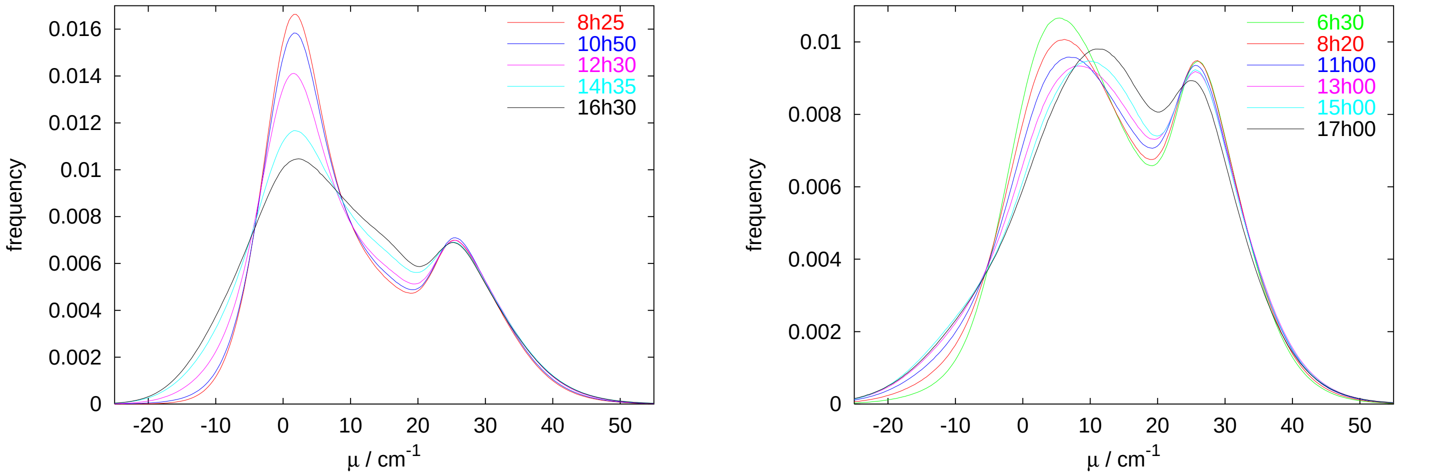

Figure 2: Histograms of the 3D images at different hardening times for a sample with $w/c = 0.97$ (left) and a sample with $w/c = 0.41$ (right).

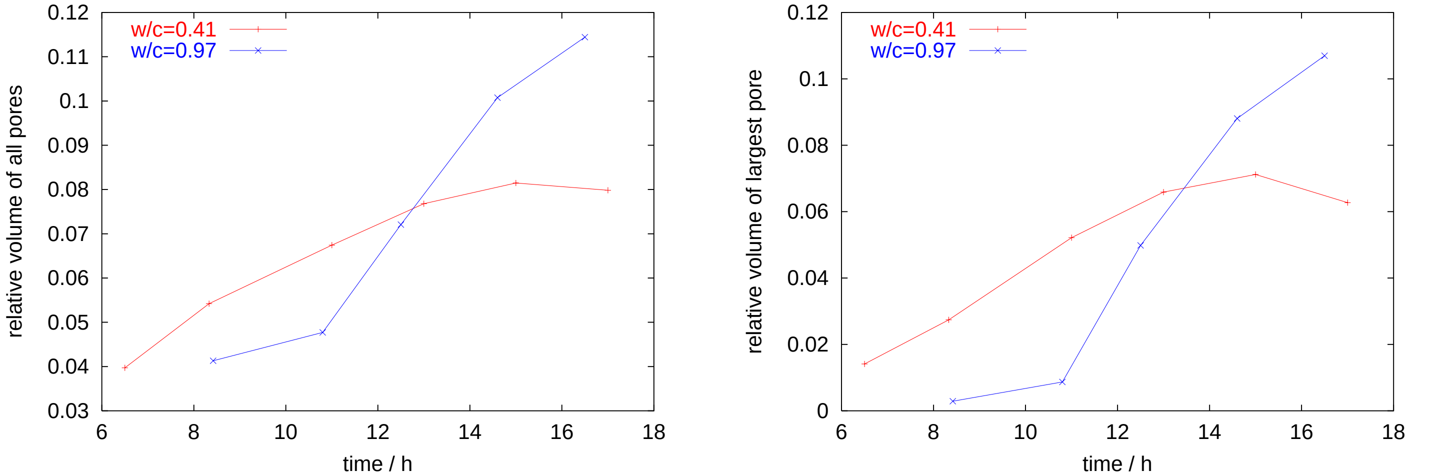

Figure 3: Total porosity (left) and relative volume (right) as a function of the hardening time for a sample with $w/c = 0.97$ and a sample with $w/c = 0.41$.

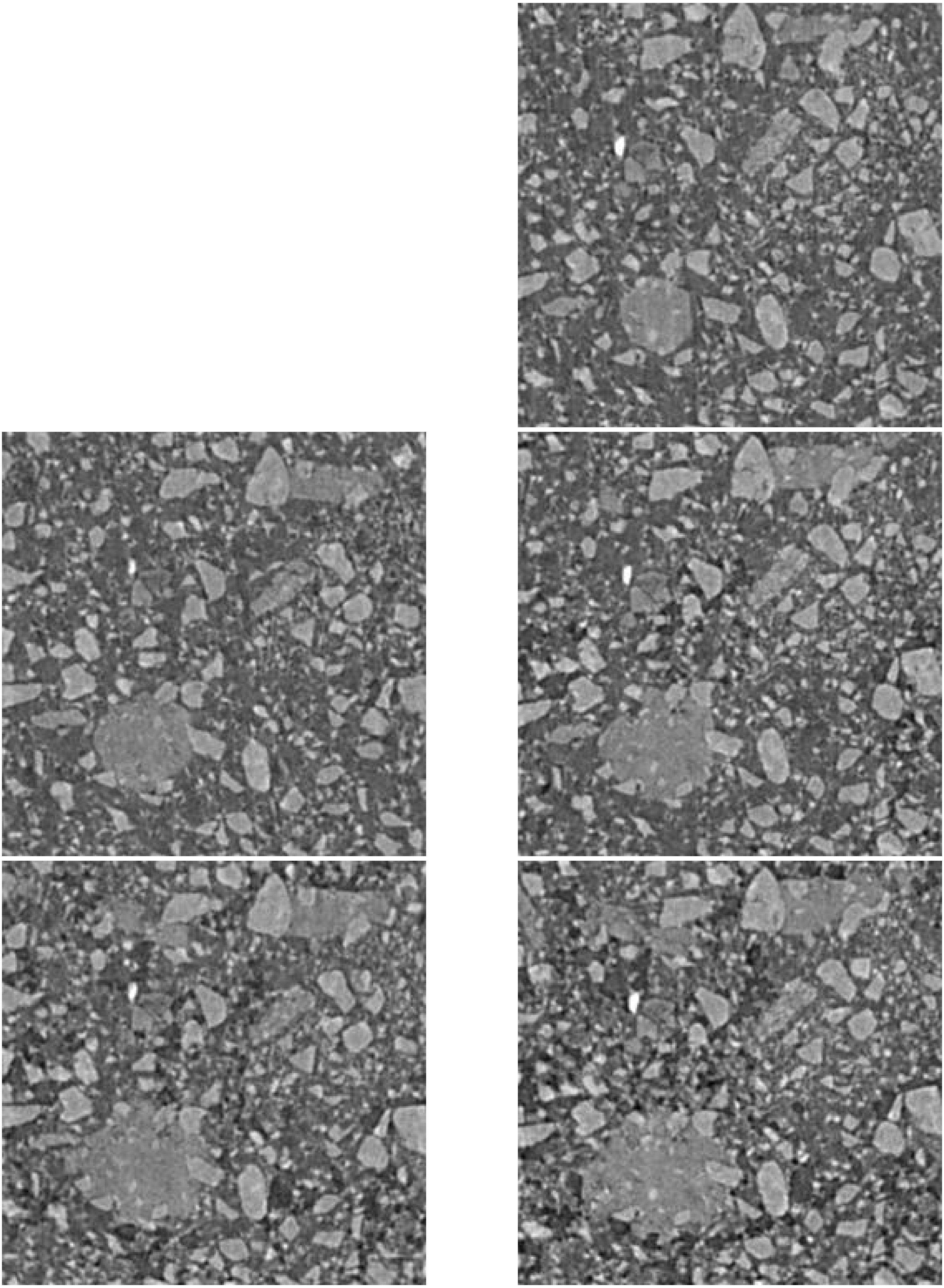

Figure 4: Cut out regions of the same tomographic slice through a sample with $w/c = 0.97$ at different solidification times: 8h25 (top right), 10h50 (centre left), 12h30 (centre right), 14h35 (bottom left), 16h30 (bottom right). Image size is 256×256 pixel. The white spot is presumably an iron-rich particle originating from the cement milling process.