THREE DIMENSIONAL CRACK DETECTION IN HARDENED CEMENT PASTES USING SYNCHROTRON-BASED COMPUTER MICROTOMOGRAPHY (SRμCT)

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ABSTRACT

Fracture patterns induced in the hardened cement pastes by instrumented indentation technique were investigated. The fracture patterns were monitored by synchrotron-based computer microtomography (SRμCT) and scanning electron microscopy. The indentation tests were carried out on specially produced microspecimens using Berkovich diamond probe. The size of the samples has been dictated by the requirements of synchrotron-based microtomography observation. The samples were of cylindrical shape and the diameter of the samples varied between 500 and 550 μm.

Samples were tomographed both before and after indentation testing. Such approach enables to reveal and possibly quantify indentation induced fracture damage reliably. The crack/damage patterns induced by indentation were also observed using environmental scanning electron microscope (ESEM). The comparison of the electron microscopy signal with 3D synchrotron-based images provides very useful information about the true size of the microcracks induced.

As the microcracks induced in the heterogeneous structure of Portland cement smear themselves into the intrinsic cement paste porosity, it is not easy to set a clear boundary between them, which makes a quantitative assessment of the indentation-induced fracture a difficult task. In spite of that a quantitative assessment of indentation induced fracture has been attempted. The outcomes of this research provides (i) very useful input for further development of micromechanical models of cement-based materials and (ii) better understanding of microfracture of cement-based materials.

1 INTRODUCTION

Synchrotron-based computer micro tomography (SRμCT) is a powerful technique for the non-destructive volumetric investigation of opaque samples. State-of-the-art SRμCT facilities provide nowadays micron- and submicron spatial resolution in a routine way [1]. In a microtomography experiment approximately 1000 of 2D X-ray projection images of a specimen are taken, while rotating the sample along its main axis. Such a set of radiographs is then mathematically reconstructed into a 3D map of the specimen absorption. Since the X-ray absorption depends directly on the composition of the object, the information held in each voxel of the 3D reconstructed image is directly proportional to the density of the material, which corresponds to such a voxel. The reconstructed 3D element is usually presented in such a manner, that the lighter the voxel is, the higher is the density of the volume element, which the voxel represents. If presented in such a manner for the pure cement paste samples, the outer air and the porosity appear dark, while the unhydrated particles are represented by the lightest voxels.

In contrary to 'surface' crack detection techniques that offer superior resolution (such as, scanning electron microscopy, atomic force microscopy), the synchrotron-based microtomography provides volumetric information about internal cracks in cement-based materials. These materials are intrinsically highly disordered and porous media and 3D information is very much required for understanding of their microfracture mechanisms caused by complex stress states. The use of SRμCT for research into fracture properties of cementitious materials is hitherto scarce. On a larger scale (6 mm-size specimens, 6 µm pixel size), fracture properties of cementitious mortars in compression were recently investigated by Landis et al. [2].The assessment of pre-critical 3D crack growth in cement is of fundamental importance for further development of micromechanical models of cementitious materials. The prime objective of the experiment reported in this paper was to find out, to what width micro-cracks induced into cementitious material by mechanical loading can still be observed using synchrotron-based computer microtomography (SRµCT).

2 SAMPLE PREPARATION

In order to realize the maximum available resolution of SRµCT, the samples must fit into the smallest available field of view of the detector (app. 720 by 720 micrometres). In fact, the sample must remain within the field of view during the whole 180° rotation. The most favourable sample form for tomography is a cylinder, since there is then the least number of artifacts in the reconstructed slices.

To comply with these requirements, cylindrical samples of pure cement paste of 500-550 µm in diameter were produced (see Figure 1). Ordinary Portland Cement, Normo 52.5R produced by Holcim, has been used. Applied water to cement ratio was equal to 0.35. The fresh cement paste was cast into specially designed Teflon moulds. These were circular sheets of 12 mm diameter and 0.5 mm height containing several (usually seven) cylindrical openings of 0.5 mm in diameter. While still embedded in Teflon moulds the faces of the cylindrical samples were ground using special grinding cloth. The samples were demoulded under an optical microscope by cutting away the Teflon moulds using razor blade. Such samples were then carefully transferred onto polished standard aluminum stub for electron microscopy by tweezers and fixed to the centre using a glue. The centre of the stub has been marked using five positioning holes of 200 µm in diameter drilled in a cross-like pattern.

3 EXPERIMENTAL

The scope of this experiment has been limited by the synchrotron beam availability. The beamtime allocated to this experiment consisted of two 8-hours long shifts, which were separated by another 8-hour long gap. This scope allowed the authors to investigate two samples. In the time between the two beamtime shifts, the samples were mechanically loaded using instrumented indentation technique.

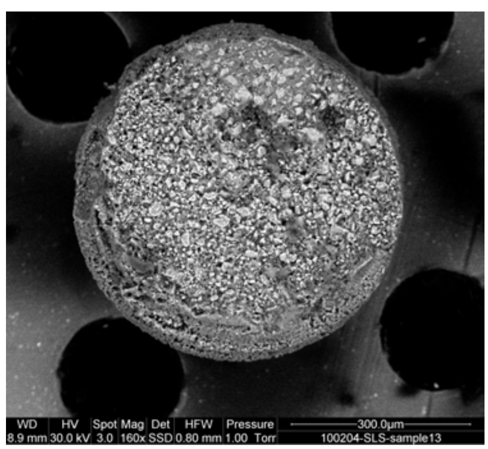

Figure 1: ESEM image of cylindrical sample of hardened OPC (diameter equals approx. 540 µm)

SRμCT has been used in pure absorption mode and the axis of the sample rotation was horizontal and perpendicular to the beam direction. Concerning other important parameters of the test arrangement, it is necessary to highlight that a Ce-doped YAG-scintillator of 20 μm thickness was used throughout this experiment. The total thickness of the scintillator was 170 μm. The distance between the sample and the scintillator was approximately 8 mm. Based on an estimation of the beam absorption in cement-based material, it was decided to use 15 keV beam energy for all the synchrotron experiments.

After being tomographed in the virgin state, the samples were transferred to environmental scanning electron microscope (ESEM), where they were subjected to mechanical loading by means of instrumented indentation. For comparison, the samples (i.e. the respective regions of interest) were observed both before and after loading. All the indentation tests performed were carried out using a standard Berkovich diamond probe and the maximum load imposed on the indenter equalled 290 mN.

After the mechanical testing was carried out, the samples were observed again using SRμCT. The identical parameters of the beam were used for the 'post-indentation' tomography experiments.

4 RESULTS

The principal result of these experiments lies in the comparison of the electron and synchrotron beam images of the region of interests (indentation sites). As for the synchrotron images, the 2D and 3D projections of indentation sites were reconstructed using Amira 3.1 software. For comparison of ESEM images with output from SRμCT, it is necessary to keep in mind the following facts: (i) the zero-tilt BSE images do not reveal the surface roughness of the sample well and (ii) the core of BSE signal comes from a certain depth below the sample surface. This depth is dependent on mean atomic number of material investigated and on accelerating voltage used. In the case of cement-based materials this depth is expected to be in single-μm range.

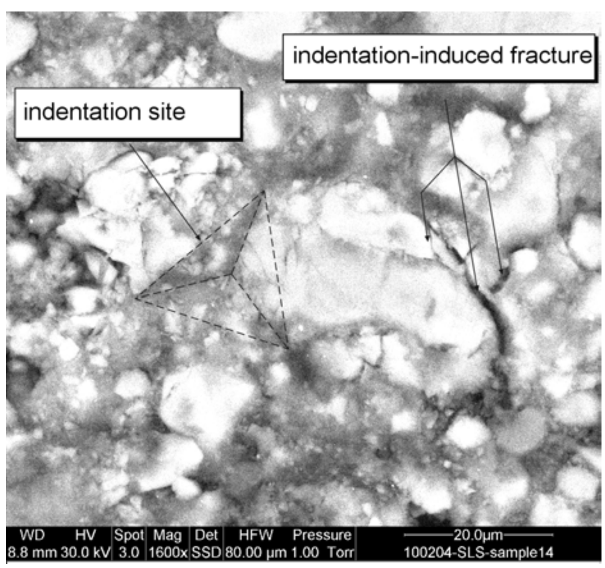

Figure 2: ESEM image of indentation site on the first sample showing the indentation-induced fracture.

Even though the Berkovich diamond probe is not the ideal indentor for fracturing the tested material, various fracture patterns in the vicinity of the indentation sites were observed using electron microscopy. Figure 2 shows a detailed BSE image of an indentation site on one of the samples tested. The width of the image is 80 µm. The size of the major crack which lies on the right-hand side of the indentation site varies between 0.8 and 1.0 µm (as measured by means of image analyses software).

For the second indentation site (see Figure 3), it is apparent that most of the fracture induced was not revealed at the surface of the specimen. Detailed ESEM image of the indentation (Figure 4) shows only minor surface cracks. However, an apparent branch-like fracture pattern can be found in the unhydrated particle positioned right below the indentation site. This can be observed in Figure 5, which presents the comparison of the corresponding reconstructed horizontal slices obtained from SRµCT. As the size of those cracks observed is in the order of several voxels only, it is hard to assess the correct size of the crack width (i.e. the crack size very much depends on the definition of its boundary). In any case, the size of the crack-width can be at least roughly estimated. The width of the crack shown below (see Figure 5) varies between 3 and 4 pixels, therefore between 1.05 and 1.4 µm. Even thinner cracks seem to be stemming from this crack, but their size is very hard to be assessed as the size approaches the pixel size of the image (i.e. 0.35 µm). A three dimensional view on this region of interest is presented in Figure 6. What becomes clear from this image is that the indentation induced fracture can be followed in all three dimensions.

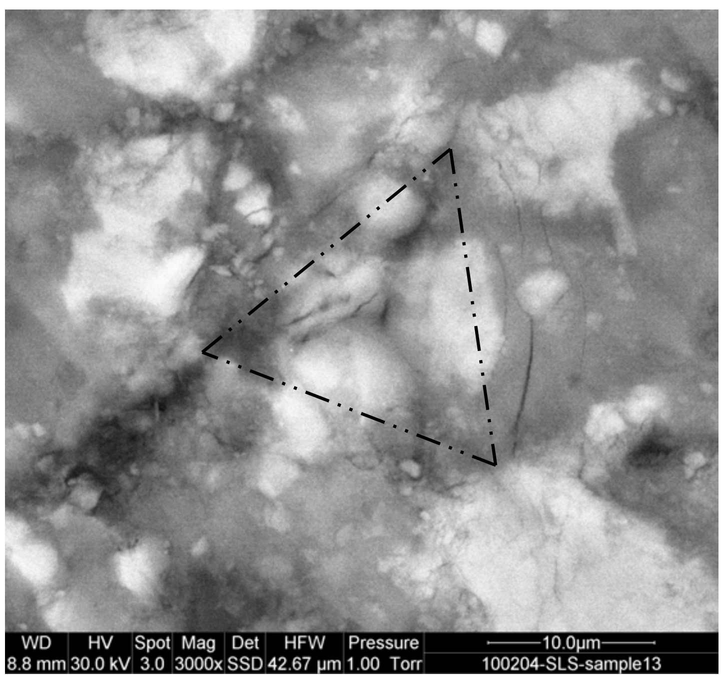

Figure 4: BSE-ESEM image of the second indentation area showing only minor surface cracks in the vicinity of the indent (0.2 to 0.3µm in width).

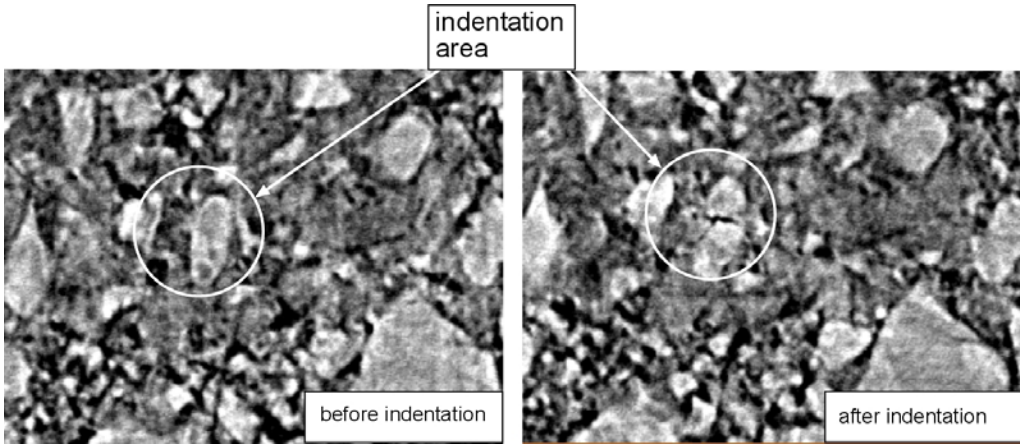

Figure 5: Two corresponding horizontal tomography reconstructed slices of the subsurface region of interest. The position of the horizontal slices is approximately 10 micrometers below the sample surface. The right image shows fracture induced by indentation.

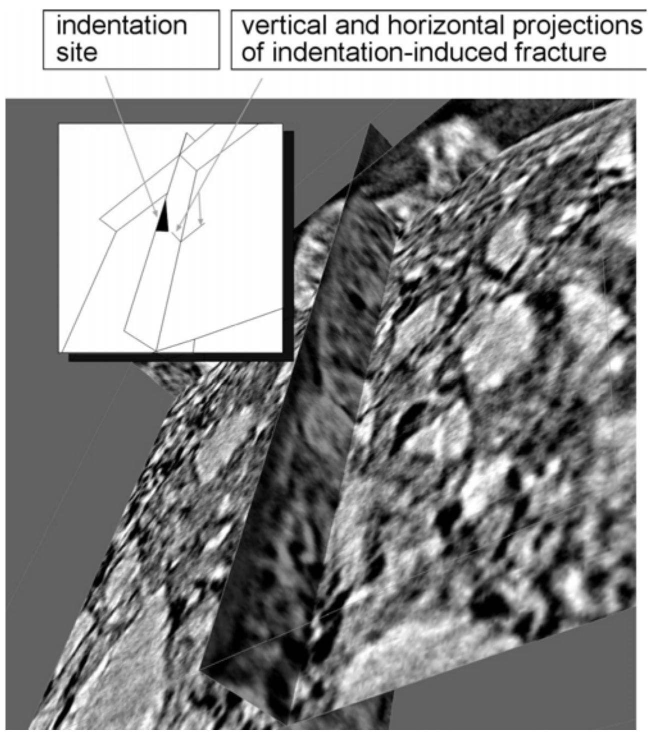

Figure 6: 3D visualization of indentation test site shown in Figure 4 by means of two orthogonal slices. The width of the indentation induced crack is approx. 3 to 4 voxels, i.e about 1 micrometre.

5 DISCUSSION AND CONCLUSIONS

Indentation using a Berkovich diamond probe was performed on microsamples of hardened ordinary Portland cement paste. Microfractures can be induced at very specific locations of the microsample, thereby allowing determining local properties of various phases in the heterogeneous microstructure of cements. From the above mentioned results, it is possible to conclude that the above mentioned experiment showed that SRμCT seems to be able to reveal cracks of about one micrometer width in cement-based materials. Above all, it is possible to follow such internal cracks in all three dimensions, in other words, to reveal non-destructively cracking below the sample surface. Indentation technique opens a path for fracture to be induced into cement-based materials with very high site specificness. An apparent disadvantage is however the complex state of stress around the indentor.

However, it becomes apparent from the SRμCT reconstructed images that it is quite hard to distinguish cracks induced by mechanical loading from intrinsic microscale porosity of cement-based materials. This becomes even more evident from images of horizontal reconstructed projections of the specimens which represent slices close to the indentation surface. There, also the influence of surface roughness plays important role. If such horizontal slices are stacked in larger numbers (approx. 10), and presented in transparent mode on coloured background, some information about the close-to-surface crack patterns can be obtained and compared with information based on ESEM images. Unfortunately, such 3D reconstructions are very hard to be reproduced in black and white only, and this is why they are not presented within this extended abstract.

6 REFERENCES

[1] Stampanoni, M., Borchert, G., Wyss, P., Abela, R., Patterson, B., Hunt, S., Vermeulen, D., Rüegsegger, P.,‘High resolution X-ray detector for synchrotron-based microtomography’, *Nucl. Inst. and Meth. A*, **491**, 291-301, (2002)

[2] Landis, E.N., Nagy, N.N., Keane, D.T., ’Microstructure and fracture in three dimensions’, *Engineering Fracture Mechanics* **70**, 911-925, (2003)