3D imaging techniques for characterising microcracks in cement-based materials

M.J. Maca, M.H.N. Yio, G. Desboisb, I. Casanovac, H.S. Wong*, N.R. Buenfeldb

a Centre for Infrastructure Materials, Department of Civil and Environmental Engineering, Imperial College London, SW7 2AZ, United Kingdom
b Structural Geology, Tectonics and Geomechanics, Energy and Mineral Resources Group, RWTH Aachen University, Germany
c Department of Civil and Environmental Engineering, Institute of Energy Technologies and Barcelona Research Center in Multiscale Science and Engineering. Universitat Politècnica de Catalunya-BarcelonaTech, Spain.

Abstract
Concrete inherently contains pores and microcracks that can adversely impact its mechanical properties and long-term durability. However, characterising microcracks is difficult due to their complex, multiscale and three-dimensional (3D) nature. This paper presents an evaluation of 3D imaging techniques for characterising microcracks induced by different mechanisms. Seven cement pastes, mortars and concretes subjected to drying shrinkage, autogenous shrinkage and freeze-thaw cycles were investigated using focused ion beam nanotomography (FIB-nt), broad ion beam serial section tomography (BIB-SST), laser scanning confocal microscopy (LSCM) combined with serial sectioning and X-ray microtomography (μCT). The study shows that the characteristics of microcracks vary significantly depending on exposure conditions. Yet there is no single technique that can capture the entire size range of microcracks from sub to tens of µm within a sufficiently representative sampling volume. The achievable image volume and resolution, and the advantages and disadvantages of each technique are compared and discussed.

Keywords: Microstructure (B); Microcracking (B); Image analysis (B); Durability (C); Concrete (E); 3D imaging

1 Introduction
Concrete as a widely used construction material is susceptible to various hygrothermal conditions that induce microcracking such as repeated wetting/drying and freezing/thawing. These inherent microcracks affect mechanical properties [1] and may facilitate ingress of deleterious substances [2] thereby initiating or exacerbating other forms of deterioration such as reinforcement corrosion. Therefore, it is essential to characterise microcracks to enable better understanding of their impact on concrete durability. However, microcracks are highly complex, multiscale and time-dependent. They vary greatly in size, density, orientation and morphology depending on the mechanism and extent of degradation. Furthermore, they propagate through cement paste, paste-aggregate interface and aggregate particles, and may ultimately form a percolated crack network.

Conventional methods for detecting microcracks in concrete structures include ultrasonic and acoustic methods. However, these provide limited information on the actual physical characteristics of microcracks. Imaging methods such as optical microscopy and scanning electron microscopy [3-6] allow direct observation and characterisation of microcracks, but they are only able to provide a two-dimensional (2D) representation of inherently three-dimensional (3D) features. Characteristics such as connectivity, tortuosity and orientation, which are relevant to mass transport, cannot be adequately determined from 2D sections [7]. Nevertheless, there exist several imaging techniques for elucidating microcracks in 3D. These can be broadly classified into destructive serial sectioning tomography and non-destructive X-ray tomography.

Focused ion beam nanotomography (FIB-nt) combines FIB milling and scanning electron microscopy (SEM) to reconstruct 3D volumes on the nm scale. Its application to cement-based materials remains limited due to the relatively small image size, typically in the order of tens of µm. Available studies have focused on characterising the particle size, shape and interfacial topology of cement particles [8-10], particle structures in cement suspension [11], surface roughness [12], nanoscale pore structure [13-18] and morphology of different solid phases [19] of hardened cement paste. Next generation Xe+ plasma FIB [19], broad ion beam serial sectioning tomography (BIB-SST) [20-22] and serial block-face SEM based on ultramicrotomy [23-25] offer possibilities to image larger volumes, potentially up to hundreds

* Corresponding author. E-mail: hong.wong@imperial.ac.uk Telephone: +44 (0)20 7594 5956
of µm in size while maintaining the resolution of SEM. These techniques are yet to be applied to investigate microcracks in cement-based materials.

Laser scanning confocal microscopy (LSCM), a form of optical sectioning microscopy, allows non-destructive 3D imaging at sub-µm resolution. This is a well-established in biological sciences, but only a handful of studies have used LSCM to investigate cement-based materials, primarily due to the limited imaging depth [26]. Examples of applications include fluorescence imaging of capillary pores, ‘Hadley’ grains and aggregate surface geometry in thin sections of mortar and concrete [26-28], real-time imaging of cement hydration [29] and surface characterisation in the reflectance mode [30-32]. Recently, LSCM has been combined with serial sectioning to reconstruct several hundreds of µm of cement-based materials at sub-µm resolution [33]. This technique has been applied to characterise and determine the representative elementary volume (REV) of capillary pores in blended cement pastes [34, 35].

X-ray micro-computed tomography (µCT) is a non-destructive 3D imaging technique, where series of 2D projection radiographs are taken at different angles to reconstruct the 3D structure of an object. This technique has seen increasing use on cement-based materials, owing to its ability to image large volumes (up to several cm thick) with minimal sample preparation. Laboratory-based µCT scanners are now capable of achieving spatial resolutions of < 1 µm, similar to those of synchrotron-based µCT [36-38]. Nevertheless, the sample size for achieving such resolutions is usually limited to sub-mm. Emerging X-ray nano-CT can achieve resolutions in the order of tens of nm in cement paste of tens to hundreds of µm thick [39, 40]. Existing applications of µCT to cement-based materials have been extensively reviewed, in particular for characterising microcracks induced by freeze-thaw [41-43], sulphate attack [44, 45], carbonation [46], and leaching [47-49].

Despite the prevalence of 3D imaging techniques and their applications to cement-based materials, no studies have focused on their feasibility for 3D characterisation of microcracks. The type and range of microcracks that can be characterised with these techniques remain unclear. This study aims to fill the gap by evaluating several 3D imaging techniques in terms of their achievable sampling volume and resolution for characterising microcracks at different length scales. FIB-n, BIB-SST, LSCM combined with serial sectioning and X-ray µCT were used to image microcracks in paste, mortar and concrete samples subjected to drying shrinkage, autogenous shrinkage, and freeze-thaw cycles. The evaluation of these 3D imaging techniques has never been carried out in a single study. The advantages and limitations of each technique, and the challenges involved are discussed. It is anticipated that the findings from this study would provide information for future research and applications concerning 3D characterisation of microcracks.

2 Experimental

2.1 Materials and samples

A range of cement paste, mortar and concrete samples with varying degrees of microcracking were prepared for investigation. Ordinary Portland cement CEM I and silica fume, with oxide composition as shown in Table 1, were used as binders. The loss on ignition (LOI), specific gravity and fineness of CEM I were 2.1%, 3.06 and 2905 cm³/g respectively. The LOI and specific gravity of silica fume were 0.47% and 2.30 respectively. CEN-Reference sand (with a maximum particle size of 2 mm), Thames Valley sand (≤ 5 mm), Thames Valley gravel (≤ 10 mm) and crushed limestone (≤ 10 mm) were used as fine and coarse aggregates. Crushed limestone (CaCO₃) was used to promote microcracking because of its higher stiffness compared to that of gravel. The particle size distributions of the aggregates are shown in Fig. 1 and their specific gravity, 24-hour water absorption and moisture content are provided in Table 2.

The mix proportions are shown in Table 3. Two cement pastes (P 0.40 & P 0.45), one mortar (M 0.50) and four concretes (C-DS, C-FT, C-AS-0.25 & C-AS-0.30) with water/binder (w/b) ratios ranging from 0.25 to 0.50 were prepared. P 0.45 and C-FT were samples from previous studies, [35] and [33] respectively. The w/b ratio of concretes containing silica fume was intentionally kept low at 0.25 to 0.30 to induce autogenous shrinkage. A polycarboxylate-based superplasticiser (Sika ViscoCrete® 20 RM) was used to improve the workability of these mixes. Additional water was added to the batch water to account for aggregate absorption in all mortar and concrete samples to ensure that the target free w/b ratios were achieved. The total aggregate volume fraction was kept in the range of 60 to 68% and the fine to total aggregate mass ratio for the concrete samples was kept at 0.4 to ensure good workability and compaction.

P 0.45 was mixed in a Hobart mixer whereas P 0.40 and M 0.50 were hand-mixed. The remaining samples were mixed in a 30-litre capacity pan mixer, where binders and aggregates were dry-mixed for 30 s prior to the addition of water for further wet mixing of 3 min. Silica fume was pre-dispersed in batching water together with superplasticiser. P 0.40 and M 0.50 were cast in plastic moulds of 30 × 30 × 15 mm³ and hand-compactend. P 0.45, C-DS and C-FT were cast in steel moulds of 100 mm diameter × 25 or 50 mm thick. C-AS-0.25 and C-AS-0.30 were cast in lidded plastic moulds of 95 mm diameter × 65 mm thick to prevent moisture exchange with the environment (to induce autogenous shrinkage). These
samples were compacted in three layers on a vibrating table until no significant release of air bubbles. All samples were compactable with no evidence of significant bleeding or segregation.

### 2.2 Curing and conditioning regimes

The samples were carefully demoulded and subjected to curing for 4, 7 or 14 days. Curing was carried out either in water, in 95% RH (fog room) or sealed in cling film at 20°C to generate varying degrees of maturity. After curing, the samples were exposed to several conditioning regimes to induce various types and degrees of microcracking. Table 4 summarises the curing and exposure conditions for all samples.

P 0.45, P 0.40, M 0.50 and C-DS were exposed to drying to generate shrinkage-induced microcracks. P 0.45 was dried at 55% RH, 20°C in the presence of soda lime to prevent carbonation, until the mass change was < 0.01% per day. P 0.40 and M 0.50 were oven-dried at 30°C for 3 d followed by solvent exchange for hydration stoppage whereas C-DS was oven-dried at 105°C.

C-FT was subjected to 56 cycles of freeze-thaw according to the test procedure described in PD CEN/TR 15177:2006 [50]. After curing in water bath, the sample was immediately surface-dried with a paper towel and placed in a freezing chamber at -20°C for 8 h. Subsequently, the sample was thawed in a water bath at +20°C for 4 h. This process was repeated for 56 cycles. Following freeze-thaw cycling, the sample was dried in a stepwise manner at gradually increasing temperatures of 30°C, 40°C and 50°C to prevent additional cracking.

C-AS-0.25 and C-AS-0.30 were kept sealed for 14 d at 20°C to generate autogenous shrinkage microcracks. There was no exchange of moisture with the environment throughout the conditioning process. Therefore, any microcracks developed in the sample could only be attributed to autogenous shrinkage caused by self-desiccation.

### 2.3 Sample preparation for microscopy

After conditioning, sub-samples were extracted from the samples and prepared for 3D imaging. The size of the sub-samples and preparation methods varied depending on the 3D imaging techniques used (Table 4). FIB-nt and BIB-SST were used to image drying shrinkage-induced microcracks in P 0.40 and M 0.50. LSCM combined with serial sectioning was used to image freeze-thaw-induced microcracks in C-FT, whereas X-ray µCT was used to image drying and autogenous shrinkage-induced microcracks in P 0.45, C-DS and C-AS.

For FIB-nt and BIB-SST, small blocks of 10 x 10 x 5 mm³ were prepared using a diamond saw. The blocks were vacuum-impregnated with low viscosity epoxy resin, ground to remove surplus epoxy and ion beam-polished to achieve a flat surface for subsequent milling and imaging. The polished surfaces were coated with a thin layer of Au to minimise charging effects under SEM imaging. For LSCM, epoxy impregnation was performed prior to the extraction of a sub-sample (40 x 20 x 8 mm³) from C-FT to preserve its microstructure. The epoxy resin was doped with 0.05 wt.% fluorescein to ensure visibility of pores and microcracks under fluorescence illumination. The block was extracted from the fully impregnated surface zone of the cylinder to ensure all microcracks and pores were filled with resin. The top face was then ground and polished down to 1 µm finish. For X-ray µCT, cylindrical cores of 400 µm, 4 mm, 9 mm and 30 mm diameter were extracted from the exposed surface of samples. Cylindrical shape was chosen because of its optimal fit within the circular field of view (FOV) of an X-ray µCT scan. There was no further sample preparation involved.

### 2.4 3D imaging

FIB-nt was performed using a Zeiss Neon 40 cross beam Ga⁺ FIB-SEM. Prior to sectioning, a region of interest (ROI) was selected and deposited with a thin layer of Pt to protect the surface from undesirable ion-induced erosion. Three trenches were then milled around the Pt layer using a 30 nA milling current to expose a cube of 20 x 20 x 20 µm³ for P 0.40 and 30 x 30 x 30 µm³ for M 0.50. Three reference lines for drift correction were created on top of the cubes using a 1 nA milling current with an exposure time of 10 s. Automated serial sectioning was then performed by repeated cycles of ion beam milling and electron imaging. Ion beam milling was performed perpendicular to the sample exposure surface whereas electron imaging was performed at an angle of 54°. A milling current of 1 nA was used and the slicing distance was set to 40 and 60 nm for P 0.40 and M 0.50 respectively. Imaging was performed in the secondary electron (SE) mode with an accelerating voltage of 2 kV and a working distance of 5.1 mm. Images were collected at a magnification of 14,650x for P 0.40 and 9,600x for M 0.50. All images were digitised to give a voxel size of 20 x 20 x 40 nm³ and 30 x 30 x 60 nm³ for P 0.40 and M 0.50 respectively. The y dimension of each voxel was corrected for distortion caused by the angle of the electron beam. Approximately 800 images were acquired for each sample and the whole process took around 15 to 20 h to complete.

BIB-SST was performed using a JEOL SM-09010 Ar⁺ ion beam cross-section polisher and a Zeiss Supra 55 SEM. The ion beam polisher was operated at 6 kV accelerating voltage with a milling current of 150 nA. A Pt masking plate was used
to guide the ion beam vertically to the sample surface to produce a cross-section of several mm². The milling process for each section took approximately 8 hours. After each milling step, the sample was coated with a thin layer of Au and transferred to the SEM for SE and BSE imaging. The microscope was operated at 25 kV accelerating voltage and a working distance of 4.6 mm. Images were collected in tile scans with a 10% frame overlap at 1,000× and 14,000× magnifications, and digitised to give a pixel size of 0.24 µm and 0.02 µm respectively.

LSCM combined with serial sectioning was performed using a Leica TCS SP5 confocal microscope and a Struers LaboPol-5 grinding machine. The method is described in detail in [33]. A Struers MD-Piano disc of 15 µm grit was used for sectioning. A 7N force was applied to the sample and grinding was carried out at 50 RPM for 2 to 3 s per direction to remove approximately 3 µm thick material. After each sectioning, a 488 nm Ar laser line at 15% intensity was used to induce fluorescence. A 40x (NA 1.25) oil immersion objective with spatial resolutions of 0.156 µm (xy) and 0.945 µm (z) was used to capture images at FOV of 350 × 350 µm². Images were digitised to a voxel size of 0.378 × 0.378 × 0.148 µm³. The sectioning and imaging process was repeated until the desired image thickness was achieved. The FOV was further extended in the xy plane by 2×2 tile scanning. The whole process took approximately 1 to 2 weeks to complete.

Four different X-ray µCT scanners with different resolving capabilities were used: an Xradia MicroXCT-400, a Metrix X-Tek HMX ST 225, an Xradia 520 Versa (Zeiss, Cambridge) and a Heliscan microCT (FEI, Houston). Each of these scanners have their respective advantages. For example, the Metrix X-Tek HMX ST 225 has a maximum operating voltage of 225 kV for improved X-ray penetration, the Xradia systems have optical lenses for additional magnification of up to 40× and the Heliscan microCT features helical scanning for stitch-free image acquisition. The imaging settings varied depending on the scanner configuration, sample type and size (Table 5). In general, the X-ray tube was operated at ≥ 100 kV to ensure sufficient X-rays penetrating through the samples. Exposure time per frame was set to ≥ 1.25 s, except for the Heliscan microCT (0.43 s for P.045 & 0.36 s for C AS_0.30 with 8 accumulations), to ensure good signal-to-noise ratio and image brightness and contrast. The total scan time ranged from ~1 to 12 h. The source-sample-detector distance was adjusted correspondingly to ensure optimal sample fit within the FOV of the detector. The resulting image voxels ranged from 200 nm to 30.2 µm. In general, the voxel size reduced with the core diameter due to ‘magnification’ of the sample in the FOV. For example, voxel sizes of 4.6 µm, 9.3 µm and 30.2 µm were achieved with core diameters of 4 mm, 9 mm and 30 mm respectively on the Xradia MicroXCT-400. In the case of Xradia 520 Versa, ‘interior’ tomography was further performed at selected ROIs with high magnification objectives. 3D volume reconstruction of the collected 2D projections was performed, with centre shift and beam hardening corrections applied.

3 Results

3.1 Focused ion beam nanotomography (FIB-nt)

The reconstructed image volumes of P 0.40 and M 0.50 acquired with FIB-nt are shown in orthogonal views and 3D renderings in Fig. 2a-d. The image volumes were drift-corrected, cropped and filtered to remove artefacts.

Drift correction was performed by aligning the image slices based on landmark (reference lines) search in the xy plane using a Template Matching plugin [52] in Fiji. An example is given in Fig. 2e showing the top view of the P 0.40 cube, where the reference lines (marked by arrows) and edges of the cube appear ‘straightened’ after alignment. Vertical stripes known as ‘curtaining’ intrinsic to FIB-nt due to variable resistance of material phases to ion milling was also observed. These artefacts were removed using a wavelet-fast Fourier transform filter as proposed by [53].

Despite the fact that the samples were impregnated with epoxy, ‘pore-backs’ [55] are visible in the reconstructed image volumes, particularly in P 0.40 (Fig. 2a-b). This could be due to uneven epoxy impregnation or over-grinding of the sample surface, resulting in voids not filled with epoxy. Nevertheless, pores and microcracks are distinguishable based on their morphology. Fig. 2a-b shows matrix and bond cracks (marked by arrows) around a solid unreacted cement grain. In 2D orthogonal views, the microcracks appear short and isolated but in 3D, they appear interconnected, long and tortuous. The measurable crack widths ranged from 0.05 to 0.5 µm, spanning across half of the image volume.

Fig. 2c-d shows the presence of microcracks in M 0.50. Only a small number of cracks were observed and they appear isolated and perpendicular to the surface in 2D and 3D. The microcracks were generally larger than those in P 0.40, ranging from 0.35 to 0.50 µm in width and < 10 µm in length.
The observed microcracks are one to two orders of magnitude smaller (width and length) than those reported in early studies involving large area 2D observation [6, 56]. The reason could be that the ROIs were sampled away from larger cracks and the image volumes were insufficient (i.e. $15 \times 12 \times 10 \, \mu m^3$ for P 0.40 and $25 \times 23 \times 20 \, \mu m^3$ for M 0.50) to capture larger cracks. Wu et al. (2015) [6] reported that mortars exposed to drying at 55%RH, 21°C and 50°C exhibited a cell-like crack pattern with crack densities of less than 1 per mm² and average crack widths and depths of 3.1 to 3.6 µm and 8.7 to 17.3 µm respectively. This indicates that the image volume required to representively capture such cracks would have to be at least a few mm in size. Clearly, FIB-nt is unable to provide such large image volumes even with Xe⁺ plasma ion sources.

### 3.2 Broad ion beam-serial section tomography (BIB-SST)

A typical BIB-polished cross section of M 0.50 is shown in Fig. 3a. The cross section was made up of 196 individual images acquired in the SE mode at 500x magnification. The milled area was pseudo-Gaussian in shape, reflecting the ion beam profile. The milled surface contained pronounced curtaining effects, similar to that observed in FIB-nt.

Furthermore, the surface suffered from non-planarity especially towards the edge due to a reduced ion flux of the beam. The total milled area was several mm², but only the centre of less than 0.5 mm² was relatively flat.

Fig. 3b presents five serial sections acquired in the centre of the milled area at higher magnifications. In the SE mode, topography effects such as curtaining and redeposition of milled materials are clearly visible. However, these effects are less obvious in the BSE mode. Distinct phases including aggregates, unreacted cement grains, hydration matrix and voids are discernible based on their greyscale and morphology.

The thickness of each serial section was measured by imaging the top of the cross section and calculating the distance between successive sections [20]. The measured inter-slice thickness ranged from 11 to 20 µm, with a standard deviation of 4.9 µm. The poor repeatability and large thickness of the sections mean that the image resolution in the z direction is low and inconsistent compared to the xy plane. Fig. 3c-e shows the reconstructed orthogonal views and 3D rendering of M 0.50. The serial sections were stacked together and aligned in Fiji. The microstructure appears elongated in the z direction due to the voxel being disproportionately anisotropic in the z plane. A significant amount of information was lost between sections. This resulted in discontinuities in the microstructure, rendering the image volume unusable for characterising microcracks.

In comparison to FIB-nt, more bond and matrix cracks, ranging from 0.5 to 2.5 µm in width (Fig. 3f, marked by arrows), were observed due to the significantly larger area imaged. Moreover, the microcracks appear longer and interconnected in 2D, spanning across hundreds of µm. The crack density seems higher than 1 per mm², with most cracks concentrating in the cement paste matrix. It is possible that some of these microcracks were caused by grinding.

### 3.3 LCSM combined with serial sectioning

Fig. 4a shows the cylinder of C-FT impregnated with fluorescent epoxy resin. It can be seen that the epoxy penetrated half of the cylinder depth, showing densely distributed cracks with significant branching on the surface, indicating severe damage caused by freeze-thaw action. Fig. 4b shows a 2D LCSM image of the cross section acquired with a 5x objective. The images (Fig. 4b-e) were converted to grey scale and inverted to highlight microcracks and pores in black. Aggregates appear as large white particles with well-defined boundaries whereas the microporous cement paste matrix fills the space in-between. Microcracks can be seen forming around the aggregate particles and through the cement paste matrix, spanning across the entire FOV.

The reconstructed single-FOV image volume is shown in Fig. 4c-d. In total, 50 image stacks with an average overlapping region of 36.6% ± 23.6% and an average phase correlation of 0.92 ± 0.04 were acquired with the 40x objective. This gave a total image volume of 302 × 302 × 167 µm³. Further details of the reconstruction are presented in [33]. Although the z resolution is 6x lower than the xy resolution, the microcracks do not appear distorted as much as the capillary pores [34] in the orthogonal views. This is because microcracks are intrinsically higher in aspect ratio and larger in size compared to capillary pores. Nevertheless, uneven brightness and discontinuities are visible along the sectioning depth.

The microcracks occupy a large fraction (~7%) of the image volume and appear highly interconnected (Fig. 4d). The main cracks branch and connect with each other via capillary pores and finer microcracks, and the crack widths vary significantly even within the same branch, from ~0.5 to 22 µm. The crack surface morphology appears highly undulated with folds and creases clearly visible.

Fig. 4e presents the extended image volume (662 × 662 × 80 µm³) with an increased FOV made up of 2 x 2 mosaic tiles, also acquired with the 40x objective. The total number of image stacks acquired was 96 and the average overlap and phase correlation between stacks were 15.5% ± 2.0% and 0.91 ± 0.05 in the xy plane and 26.9% ± 10.4% and 0.88 ± 0.04 in the z plane respectively. Due to the larger FOV, the propagation of cracks around aggregates and through the paste...
matrix were more representatively captured. It can be clearly seen that the bond cracks surrounding aggregates tend to be larger than those in the paste matrix, suggesting that the cracks have initiated at the aggregate-paste interface. Moreover, many cracks which appear isolated in 2D are interconnected via either smaller cracks or pores, forming a percolated network in 3D. In comparison with drying shrinkage-induced microcracks [6], freeze-thaw-induced microcracks appear more interconnected, tortuous, densely distributed and variable in width.

The smallest crack widths observed were one to two orders of magnitude finer than those reported in [41, 43], which used X-ray μCT to investigate mortars exposed to freeze-thaw action. This indicates that a significant number of smaller cracks might have been missed in the studies although the image volumes were 2,600× to 7,000× larger than that above. It is possible to extend the image volume of LSCM to the order of cm³ but the acquisition process would be significantly prolonged.

### 3.4 X-ray microtomography (μCT)

The effect of sample size on voxel resolution of X-ray μCT is demonstrated with C-DS cores in Fig. 5a-b. Similar to BSE images, pores and microcracks appear the darkest whereas unreacted cement particles (< tens of µm) appear brightest. As the core diameter decreased, phase contrast between fine aggregates and cement paste improved due to increased differential X-ray absorption.

The detectable crack widths in C-DS ranged from ~0.4 to 16 µm, ~0.4 to 68 µm and ~0.4 to 115 µm in the 4 mm, 9 mm and 30 mm cores respectively due to the reduction in voxel resolution (Table 5). Nevertheless, there were not many cracks and most were bond cracks surrounding aggregate particles, appearing isolated with no specific orientation. It is likely that the bond cracks are interconnected via finer matrix cracks, as observed in P 0.40 and M 0.50 with FIB-nt and BIB-SST, but not adequately resolved with X-ray μCT. Indeed, the smallest cracks observed were two to three orders of magnitude larger than those measured by [6] on a similar w/c 0.5 concrete dried at 105°C, in which more than 80% of the cracks were < 10 µm. This suggests that a significant proportion of microcracks is not detected with X-ray μCT.

The C-AS-0.25 and C-AS-0.30 cores (30 mm diameter) scanned with Metris X-Tek HMX ST 225, Heliscan microCT and Xradia 520 Versa achieved higher voxel resolutions of 15.5 µm, 12.7 µm and 22.5 µm respectively (Fig. 6a) due to higher detector efficiencies. In any case, these voxel resolutions are no better than the actual spatial resolution of the scanners and the minimum resolvable feature remains 2× to 3× the voxel size (see Discussion). Despite differences in voxel resolution, the samples appear similar in terms of the extent and morphology of microcracking. The Heliscan microCT gave the smallest detectable crack widths amongst the three systems. Nevertheless, the interior tomography obtained with Xradia 520 XRFM at voxel resolution 3 µm revealed the presence of smaller microcracks (~10 µm) although the FOV and signal-to-noise ratio were significantly poorer.

In comparison to C-DS, the cores from C-AS showed higher degree of microcracking, with crack widths ~37 to 90 µm. Furthermore, the microcracks formed preferentially through air voids, aggregate-paste interface and cement paste matrix, suggesting that the air voids facilitate crack propagation. Fig. 6b shows an orthogonal view of the C-AS-0.30 core acquired with Heliscan microCT. It can be seen that the microcracks exhibit preferential orientation parallel to the xy plane, indicating strong anisotropy in 3D. This was not observed in the section views (Fig. 6a). The orientation of cracks may be related to sample shape and dimension, orientation of aggregates, casting direction and compaction. Fig. 6c shows the microcrack network and air voids in C-AS-0.25. The microcracks appear highly connected, tortuous and densely distributed throughout the entire image volume. Many spherical entrapped air voids were also observed, due to incomplete compaction of the low w/c ratio (0.25) concrete. Further quantification of the microcracks and air voids in terms of volume fraction, density and size will be available in a separate publication [58].

Further reduction in voxel resolution is possible with smaller sample sizes. Fig. 7a-d shows example images of P 0.45 cores of 4 mm and 400 µm diameters acquired with the Heliscan microCT and Xradia 520 Versa with voxel resolutions of 2.29 µm and 0.2 to 0.4 µm respectively. The images acquired with Heliscan microCT (Fig. 7a-b) show a homogenous and dense paste microstructure. Unreacted cement particles with well-defined morphology can be seen evenly distributed throughout the sample. A microcrack of ~4 µm width, probably caused by coring, is also visible (Fig. 7b, marked by arrows). Nevertheless, capillary pores were not observed due to insufficient resolution. In contrast, the 3D images obtained with Xradia 520 Versa (Fig. 7c-d) appear more porous. A cracked unreacted cement particle is seen in Fig. 7d. Nevertheless, the spatial resolution is inferior to those of FIB-nt, BIB-SST and LSCM.
4 Discussion

4.1 Challenges in 3D imaging of microcracks

Our results show that important characteristics of microcracks including connectivity, orientation and length cannot be adequately characterised in 2D. Although 3D imaging provides better visualisation and characterisation, this remains a challenging task as the size of microcracks spans from sub-μm to hundreds of μm, and their morphology and density vary depending on the damage mechanism.

Some general observations can be drawn on the microcracks investigated in this study. Drying shrinkage-induced microcracks are low-density and multiscale, ranging from sub to hundreds of μm depending on drying severity and presence of aggregates. Autogenous shrinkage produces microcracks of tens of μm wide that are densely distributed and preferentially orientated. Freeze-thaw cycles generate microcracks in the range of sub to tens of μm, which are well-distributed and highly interconnected. Clearly, the image volume and resolution required for capturing these cracks depend on the crack width and extent of cracking.

For a typical concrete with a maximum aggregate size of 10 or 20 mm, the image volume should be in the order of several cm to ensure that microcracks around aggregate particles are captured. The required image resolution depends on the crack width, ideally sub-μm to capture the finest microcracks. Nevertheless, there is yet to be a technique capable of achieving such resolution on a cm-scale image volume. The REV for characterising microcracks remains to be established. However, 3D numerical modelling of microcracked concrete [59] suggests that a computational cube 2.5× the largest aggregate particle can give representative mass transport provided that a sufficient number of replicates is simulated and the results averaged. Interestingly, experimental studies have also shown that an image volume of ~ 20 × 20 × 25 mm³ is adequate for quantifying most parameters of autogenous shrinkage-induced microcracks in concrete[57, 58]. This is significantly larger than the REV for capillary pores, which is in the order of 10³ μm³ [34].

4.2 Serial sectioning methods

Serial sectioning and imaging methods such as FIB-nt, BIB-SST and LSCM require a series of sample preparation steps (drying, epoxy impregnation, grinding and polishing) that may damage the sample and introduce artificial cracks, further complicating analyses. The sectioning process can also generate artefacts such as curtaining, scratches, redeposition of materials and non-planar sections. Furthermore, the lengthy imaging process may cause variations in image resolution, brightness and contrast. All these factors complicate the reconstruction and subsequent analysis of the image dataset. Nevertheless, they can be minimised by careful execution and image processing.

Another limitation intrinsic to serial sectioning is anisotropic resolution of the acquired image dataset. This occurs because the resolution in the sectioning direction tends to be worse than that of the imaging plane. This can result in image distortion and prevent orientational characterisation of microcracks. In the case of FIB-nt and BIB-SST, the axial resolution is directly related to the thickness of serial sections. For FIB-nt, the voxel dimension can be adjusted to achieve isotropy thanks to its ability to produce uniform nm thick sections. For BIB-SST, the minimum achievable section thickness reported to date is 260 nm [22] which is much finer than those achieved in this study. Nevertheless, it remains difficult to ensure consistently uniform and parallel sections. For LSCM, axial distortion occurs due to the elongated nature of the point spread function and mismatch of refractive indices between the sample and immersion liquid. Axial compression based on the known shape or size of reference materials can be applied to correct for this effect [34, 60].

Perhaps, one of the biggest disadvantages of serial sectioning is their inability to ‘pre-screen’ the internal structure of a sample. As such, the selection of a suitable ROI for 3D imaging is a hit and miss process. This would be problematic for features that are highly variable such as microcracks. Uneven or inhomogeneous epoxy impregnation can further complicate the process. For LSCM, insufficient epoxy impregnation may cause some pores and microcracks to be undetected. Serial sectioning methods are also destructive, meaning the ROI cannot be revisited once sectioned. As such, it would highly desirable to be able to pre-select the ROI. This can be achieved with the use of non-destructive X-ray μCT to obtain an overview of the internal structure prior to sectioning.

4.3 Comparison of 3D imaging techniques

Fig. 8 presents a graphical summary of the operating regimes of the 3D imaging techniques. The data points were derived from the present study and from literature. The y-axis represents the limiting spatial resolution calculated as 2.3× the longest dimension of the achievable voxel based on the Nyquist theorem [61]. The x-axis indicates the achievable thickness of the image volume (not the actual sample physical dimension). In the case of FIB-nt, BIB-SST and LSCM, the image resolution and thickness are determined from the thickness (axial resolution) and extent of serial sectioning respectively.
It is clear from the figure that resolution reduces with increasing image thickness irrespective of the imaging techniques and that none of the techniques are capable of covering the entire range of microcracks. LSCM bridges the gap between nanoscale (FIB-nt & BIB-SST) techniques and X-ray µCT. New techniques such as X-ray nanoCT [39, 40] and Xe+ plasma FIB-nt [19] enable image thickness of up to ~100 µm and resolutions of ~0.1 µm. FIB-nt is limited by the achievable image volume (tens of µm) because the ion milling process is slow and accumulates artefacts. As such, it is suitable only for localised characterisation of microcracks for applications such as fracture surface analysis.

BIB-SST is able to provide much larger image area than FIB-nt (up to mm²), but the image thickness and resolution (section thickness) is limited by the manual sectioning and imaging process. Other studies [21, 22] suggest the possibility of achieving sub-µm section thickness with BIB but the section remains inconsistent and non-planar. Perhaps, the real advantage of this technique can only be realised when the entire process becomes fully automated. Nevertheless, BIB-SST is useful for large-area observation of microcrack networks in 2D.

The image volume of LSCM can, in theory, be extended to cm-scale to capture microcracks in mortar or concrete samples without degradation in resolution. However, the laborious sectioning and imaging process limits the technique for routine use. With a low-power objective (e.g. 10× NA 0.30), it may be possible to reconstruct image volume as large as 7 × 7 × 2 mm³ with axial resolution of ≥10 µm [62]. Furthermore, unlike the other techniques, LSCM detects only fluorescence in capillary pores and microcracks, and does not distinguish solid phases.

For X-ray µCT, there is an inevitable trade-off between achievable resolution and image volume. Its resolution degrades from a few µm to tens of µm as the image volume increases from mm to cm. On the one hand, a large proportion of fine microcracks (<10 µm) such as those induced by drying shrinkage will be excluded if imaging is carried out at cm scale. On the other hand, imaging at mm scale to resolve microcracks may not be sufficiently representative.

One may be tempted to compare 3D imaging techniques with other indirect techniques such as mercury intrusion porosimetry (MIP) and gas adsorption. However, these techniques are limited to pastes and mortars. Furthermore, they are incapable of distinguishing between microcracks, capillary pores, and air voids. As such, it would be impossible to make meaningful comparisons between them.

### 4.4 Alternative approaches

'Hierarchical' imaging combines complementary techniques to image at varying resolutions and image volumes without destroying the sample. X-ray µCT with adjustable FOVs [63] is used in conjunction with precise positioning based on landmarks [64] to identify ROIs for subsequent imaging with higher resolution techniques such as synchrotron-based X-ray CT. The ROIs may be stitched together to form larger image volumes [65]. However, a prerequisite of this approach is that the sample is resolvable at different length scales independent of the physical sample size.

The high resolution interior tomography on the Xradia 520 Versa X-ray CT is an example of hierarchical imaging. But as shown in this study, the very dense nature of concrete limits this approach and it is not possible to achieve sub-µm resolution without reducing the actual physical sample size to <1 mm. However, with rapid advancement in high powered X-ray sources and larger flat panel detectors [66], it may soon be possible to achieve three or more decades of resolution within a fixed sample size.

Another approach, but destructive, is to subdivide the sample to smaller volumes for separate X-ray imaging at high resolution. The sub-volumes are then stitched together to produce a final 3D dataset. A global overview of the sample can be pre-acquired at low resolution to facilitate the reconstruction process. A similar approach has been applied to reconstruct large volumes of neural tissues (~120 × 32 × 80 µm³) with FIB-nt at a voxel resolution of 8 × 8 × 8 nm³ [67], using hot-knife sectioning or ultramicromtome to split tissues into 20 µm thick sub-volumes. In theory, such approach is applicable to cement-based materials. But a precise sectioning method that yields clean cuts on hard brittle materials with minimal material loss between sub-volumes does not yet exist.

A statistical approach [68] involving sub-sampling of a large number of image volumes smaller than the REV is another possible approach. This has been applied to computationally generated cracks to model their impact on transport properties of concrete [59, 69]. However, the validity of such approach for quantifying the topology of real cracks including length, connectivity and tortuosity remains questionable. The difficulty lies in the fact that real cracks propagate and form highly interconnected networks which are multiscale, heterogeneous and anisotropic. In addition, microcracks are dependent on the size and orientation of aggregate particles. Therefore, statistical sub-sampling may lead to misrepresentation of these parameters.
5 Conclusions

We evaluated 3D imaging techniques for their ability to characterise microcracks in cement-based materials. These included focused ion beam nanotomography (FIB-nt), broad ion beam serial section tomography (BIB-SST), laser scanning confocal microscopy (LSCM) combined with serial sectioning and X-ray microtomography (µCT). The evaluation of these advanced techniques has never been carried out in a single study. Seven cement pastes, mortars and concretes subjected to drying shrinkage, autogenous shrinkage and freeze-thaw cycles were examined. The key findings are:

a) 3D imaging provides information which cannot be readily obtained from 2D images including microcrack connectivity, length and orientation. Microcracks are highly multiscale and morphologically variable depending on the degradation mechanism. Autogenous shrinkage and freeze-thaw cycles produce microcracks which are more interconnected and higher in density than those produced by drying shrinkage.

b) FIB-nt provides nm-scale resolution, but the image volume is limited to tens of µm. As such, FIB-nt is more appropriate for localised characterisation of pores and microcracks. BIB-SST can provide larger image areas (a few mm²) but the serial sections are relatively thick (10 to 20 µm), inconsistent and non-parallel, resulting in discontinuities in microcrack images.

c) LSCM combined with serial sectioning provides image volumes of several hundreds of µm with sub-µm resolution. This is sufficient to characterise capillary pores at representative elementary volume (REV). The image volume can be further extended to fully capture microcracks around aggregates in mortar or concrete by means of tile imaging, but the process is time-consuming and laborious.

d) X-ray µCT is non-destructive and hence advantageous for 3D characterisation of large pores and microcracks. However, its resolution is highly dependent on sample size. For cm-scale samples, the minimum resolvable microcrack is ~10 µm. Smaller microcracks are observable only in mm-scale samples. Its spatial resolution is inferior to those of FIB-nt, BIB-SST and LSCM.

e) Destructive techniques including FIB-nt, BIB-SST and LSCM combined with serial sectioning may introduce artefacts such as curtaining, scratches, material redeposition and artificial microcracks. Furthermore, these techniques tend to have anisotropic voxel, which may pose problems for orientational characterisation. In addition, destructive techniques are unable to ‘pre-screen’ the internal structures for accurate selection of ROIs.

f) None of the available 3D imaging techniques can cover the entire range of microcracks in concrete at sufficiently high resolution and achieve a representative sampling volume. However, in combination, they enable localised and global characterisation of microcracks.

g) A graphical summary of achievable spatial resolution against image thickness for 3D imaging techniques has been produced based on data from this study and those available in the literature (Fig. 8). Resolution decreases with increasing image thickness irrespective of the imaging technique. It is anticipated that this master plot is useful for informing future research and applications concerning 3D characterisation of microstructure.
Acknowledgements

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References


# 3D imaging techniques for characterising microcracks in cement-based materials

M.J. Mac, M.H.N. Yio, G. Desbois, I. Casanova, H.S. Wong, N.R. Buenfeld

## Table 1: Oxide composition of binders.

<table>
<thead>
<tr>
<th>Materials</th>
<th>CaO</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
<th>MgO</th>
<th>Na$_2$O</th>
<th>K$_2$O</th>
<th>SO$_3$</th>
<th>Cl$^-$</th>
<th>TiO$_2$</th>
<th>P$_2$O$_5$</th>
<th>Mn$_2$O$_3$</th>
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<tr>
<td>CEM I</td>
<td>63.4</td>
<td>20.6</td>
<td>5.6</td>
<td>2.4</td>
<td>1.6</td>
<td>0.2</td>
<td>0.7</td>
<td>2.9</td>
<td>&lt; 0.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>Silica fume</td>
<td>0.15</td>
<td>98.7</td>
<td>0.31</td>
<td>0.02</td>
<td>0.04</td>
<td>0.09</td>
<td>0.30</td>
<td>-</td>
<td>0.39</td>
<td>0.02</td>
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## Table 2: Properties of aggregates.

<table>
<thead>
<tr>
<th>Aggregate type</th>
<th>Max aggregate size (mm)</th>
<th>Specific gravity</th>
<th>24-hr absorption (%)</th>
<th>Moisture content (%)</th>
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<tbody>
<tr>
<td>Sand</td>
<td>5</td>
<td>2.56</td>
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<tr>
<td>Gravel</td>
<td>10</td>
<td>2.70</td>
<td>0.76</td>
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<tr>
<td>Limestone</td>
<td>10</td>
<td>2.71</td>
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</table>

## Table 3: Mix proportions.

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>CEM I (kg/m$^3$)</th>
<th>Silica fume (kg/m$^3$)</th>
<th>Water (kg/m$^3$)</th>
<th>Free w/b$^*$</th>
<th>Sand (kg/m$^3$)</th>
<th>Gravel (kg/m$^3$)</th>
<th>Limestone (kg/m$^3$)</th>
<th>Aggregate volume fraction (%)</th>
<th>Superplasticiser (kg/m$^3$)</th>
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<tr>
<td>P 0.40</td>
<td>1376</td>
<td>-</td>
<td>550</td>
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<tr>
<td>P 0.45</td>
<td>1290</td>
<td>-</td>
<td>581</td>
<td>0.43</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>M 0.50</td>
<td>510</td>
<td>-</td>
<td>255</td>
<td>0.50</td>
<td>1531</td>
<td>-</td>
<td>67</td>
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<tr>
<td>C-DS</td>
<td>399</td>
<td>-</td>
<td>200</td>
<td>0.50</td>
<td>702</td>
<td>-</td>
<td>1053</td>
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<td>194</td>
<td>0.50</td>
<td>712</td>
<td>1068</td>
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<td>67</td>
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<td>C-AS-0.25</td>
<td>488</td>
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<td>712</td>
<td>1068</td>
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<td>60</td>
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<tr>
<td>C-AS-0.30</td>
<td>451</td>
<td>45</td>
<td>149</td>
<td>0.30</td>
<td>718</td>
<td>1077</td>
<td>-</td>
<td>68</td>
<td>5</td>
</tr>
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</table>

*Corrected for bleeding

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1 Corresponding author. E-mail: hong.wong@imperial.ac.uk Telephone: +44 (0)20 7594 5956
**Table 4: Curing and exposure conditions and imaging methods for all samples.**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample dimensions (mm)</th>
<th>Curing age (d)</th>
<th>Curing condition</th>
<th>Exposure condition</th>
<th>Imaging method</th>
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<td>P 0.40</td>
<td>30 × 30 × 15</td>
<td>4</td>
<td>95 ± 2% RH</td>
<td>Oven drying at 30°C</td>
<td>FIB-nt</td>
</tr>
<tr>
<td>P 0.45</td>
<td>100 Ø × 25</td>
<td>7</td>
<td>95 ± 2% RH</td>
<td>55% RH at 20°C</td>
<td>X-ray µCT</td>
</tr>
<tr>
<td>M 0.50</td>
<td>30 × 30 × 15</td>
<td>4</td>
<td>95 ± 2% RH</td>
<td>Oven drying at 30°C</td>
<td>FIB-nt, BIB-SST</td>
</tr>
<tr>
<td>C-DS</td>
<td>100 Ø × 50</td>
<td>7</td>
<td>95 ± 2% RH</td>
<td>Oven drying at 105°C</td>
<td>X-ray µCT</td>
</tr>
<tr>
<td>C-FT</td>
<td>100 Ø × 50</td>
<td>14</td>
<td>In water</td>
<td>56 freeze-thaw cycles</td>
<td>LSCM</td>
</tr>
<tr>
<td>C-AS-0.25</td>
<td>95 Ø × 65</td>
<td>14</td>
<td>Sealed</td>
<td>Sealed at 20°C</td>
<td>X-ray µCT</td>
</tr>
<tr>
<td>C-AS-0.30</td>
<td>95 Ø × 65</td>
<td>14</td>
<td>Sealed</td>
<td>Sealed at 20°C</td>
<td>X-ray µCT</td>
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**Table 5: X-ray µCT imaging settings.**

<table>
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<tr>
<th>Sample</th>
<th>Xradia MicroXCT-400</th>
<th>Metris X-Tek HMX ST 225</th>
<th>Xradia 520 XRM</th>
<th>Heliscan microCT</th>
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<td>C-DS</td>
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Fig. 1: Particle size distribution of aggregates.
Fig. 2: FIB-nt imaging results. a-d, Orthogonal views and 3D renderings of the reconstructed image volumes of P 0.40 cement paste (a-b) and M 0.50 mortar (c-d). Dimensions in µm. e, Alignment of the image slices based on reference lines. f, Removal of curtaining effect induced by ion-milling by wavelet-fast Fourier transform filter. g, Greyscale variation along image width of (f) before and after filtering.
Fig. 3: BIB-SST imaging results of M 0.50 mortar. a, Milled area revealing the pseudo-Gaussian beam profile. b, Successive milled sections acquired in BSE and SE modes. c-d, Orthogonal views (c) and 3D renderings (d) of the reconstructed image volume in BSE mode. Dimensions in µm. e-f, Orthogonal views in SE mode (e) showing microcracks in a close-up (f).
Fig. 4: LSCM imaging results of C-FT concrete. a, Microcracks on the cylinder surface revealed by epoxy impregnation. b, 2D overview of the microcracks acquired with a 5x objective. c-f, Orthogonal views (c, e) and 3D renderings (d, f) of the reconstructed single-FOV (c-d) and extended (e-f) image volumes acquired with a 40x objective. All dimensions in µm.
Fig. 5: X-ray μCT images of C-DS cores of 30 mm, 9 mm and 4 mm diameter acquired with Xradia MicroXCT-400. a, Example 2D image slices showing detectable microcracks (marked by arrows). b, Corresponding 3D renderings. All dimensions in µm.
Fig. 6: X-ray µCT images of C-AS cores. a, Comparison of imaging results across different scanners. b, Orthogonal view of C-AS-0.30 core. c, Extracted microcrack network and air voids from the reconstructed image volume of C-AS-0.25 core. All dimensions in µm.
Fig. 7: X-ray μCT images of P 0.45 cores. a, Example 2D image slice of a 4 mm diameter core acquired with FEI Heliscan microCT. b, Close-up of a microcrack in (a). c, 3D rendering of a 400 μm core imaged with Xradia 520 XRM. Dimensions in μm. d, Example 2D image slice from the interior tomographic volume in (c).
Fig. 8: Comparison of 3D imaging techniques in terms of achievable image thickness and spatial resolution. Circles (○) denote data for FIB-nt, squares (□) for BIB-SST, crosses (×) for X-ray nanoCT, triangles (△) for LSCM and diamonds (◇) for X-ray µCT. Empty and filled symbols represent data from present study and literature respectively. Operating regimes of Xe+ PFIB-nt and X-ray nanoCT are adapted from [19].