X-ray dynamic observation of the evolution of the fracture process zone in a quasi-brittle specimen

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ABSTRACT: The aim of this work is the evaluation of the fracture process zone while loading a quasi-brittle concrete compound. The regularly used optical observation of the specimen surface does not provide accurate information regarding the fracture zone shape, particularly when this zone is tunnelled inside of the specimen body. Therefore, X-ray dynamic deflectoscopy and computed tomography were employed as tools for an extended investigation of process zone evolution. A notched specimen manufactured from silicate composite was subjected to the three-point bending test in a special table-top loading device. On-line radiographic observation of the process zone during the loading experiment serves for overall evaluation, while a tomographic measurement - which is conducted during temporal loading interruption - provides information about the spatial distribution of the newly developed cracks.

KEYWORDS: Inspection with x-rays; Detection of defects

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1 Introduction

During the fracture of quasi-brittle and ductile materials, energy dissipation takes place in an extensive area at the crack tip — a nonlinear fracture process zone [1]. The aim of this work is to evaluate fracture process zone (FPZ) evolution during a loading experiment. It is well known that the FPZ is often tunnelled and branched inside of the specimen body, although its outer surface can remain unaffected. This behaviour is prevalently induced by the specimen’s geometry and by the loading type [2–7]. Since false conclusions about FPZ evolution can be deduced utilizing traditional optical observation of the specimen surface, a more sophisticated experimental method has to be employed for this purpose such as acoustic emission analysis [8–11], holographic interferometry, infrared thermography or X-ray imaging.

In this work, the techniques of time-lapse X-ray dynamic defectoscopy [12, 13] and computed tomography are utilized to obtain detailed information about FPZ evolution. The results will also serve as a reference for other types of loading, such as split tests, for instance. A notched silicate composite specimen was subjected to a three-point bending test using a special table-top loading device [14]. The overall FPZ evolution was observed radiographically on-line while increasing loading until specimen rupture occurred. Computed tomography data were recorded at the characteristic points of the load-displacement diagram, and a spatial distribution of the FPZ was analysed.

2 Experimental

A block shaped specimen with dimensions h/w/d 10.0/37.5/20.0 mm was prepared from a silicate composite (type of the concrete) exhibiting ductile behaviour. A side notch was cut in the sample using a diamond saw with a blade of 2.6 mm nominal thickness and a circular tip.
2.1 Table-top loading device

A three-point bending test was carried out using an in-house designed table-top loading device [14]. The device was specifically designed for mechanical testing with simultaneous X-ray observation of the specimen structure during loading. For this purpose, the cylindrical chamber device accommodating the specimen was manufactured from high-strength, reinforced composite carbon fibre with 0.6 mm thickness (for low X-ray attenuation). The specimen chamber was connected via a bayonet lock with the upper part of the loading device made of a high-performance aluminium alloy. The upper part of the frame contained the loading mechanism (high precision ball screw, zero backlash harmonic drive and a stepper motor) and a loading platen equipped with a load-cell with 10 kN loading capacity. The rigid behaviour of the load-cell (higher loading capacity provides for higher rigidity of the load-cell body) is an important parameter for the testing of quasi-brittle materials, as it prevents additional quasi-dynamic loading and the possible rupture of the specimen caused by stored elastic energy.

The loading platen and rounded supports of the three-point bending setup were manufactured from a carbon fibre composite allowing X-ray inspection of the material’s behaviour in the vicinity of these supports. To ensure a high quality of the CT measurement, and to simultaneously minimize the influence of the setup geometry on FPZ evolution, the distance of lower supports was set to 30 mm. The experiment was displacement-driven with a constant loading velocity of 0.5 $\mu$m/s. Such a low velocity was maintained thanks to a harmonic gearbox with zero backlash and a ratio of 1:100 connected to the precious ground ball screw with a 5 mm pitch. The stepper motor rotation was smoothed using a micro-stepping feature of the controller (10 microsteps per 1 full step). Micro-stepping cannot be used for reaching higher precision but only for reaching sufficient speed stability (20 micro-steps/s), and it works well (without micro-step loss) up to approx. 1 kN (10% maximum load capacity). Movement accuracy of the overall loading system was experimentally determined to be better than 5 $\mu$m and was controlled by computer numerical control (CNC) software. The specimen is depicted in figure 1 and the experimental device in figure 2. The X-ray image of the loading device chamber containing the specimen with the three-point bending setup is shown in figure 3.

![Figure 1. Dimensions of the silicate composite specimen.](image)
2.2 Modular dual source tomographic scanner

A modular dual-source tomographic scanner was employed for scanning of the specimen during the three-point bending test. A standard cone-beam tomography arrangement with a single X-ray tube (XWT-240-SE, X-Ray WorX, Germany) and a flat panel detector XRD1622 (Perkin Elmer, U.S.A.) with an active area of $41 \times 41$ cm, a pixel matrix of $2048 \times 2048$, and a resolution of 200 $\mu$m was utilized for observation of the loading experiment. The loading device was placed on a high-precision rotation stage APR-150 (Aerotech, U.S.A.). X-ray setup is shown in figure 4.

The X-ray setup geometry was adjusted at a focus-detector distance of 1335 mm and a focus-object distance of 147 mm, resulting in 9.1x magnification and a 22 $\mu$m pixel size. The X-ray tube was operated at 120 kV voltage and 200 $\mu$A target current. The acquisition time of the detector was set at 1 s to minimize tomography scanning time and material relaxation. Similarly, only 400 angular projections were taken in each tomography scan for these reasons (relaxation is represented by the local drop downs of the load-displacement diagram, see figure 5). Using digital image correlation technique (DIC) it was proven that the shift induced by material relaxation was only 3 $\mu$m (approx. 14% of pixel size), and this didn’t affect the tomographic measurement (precision of used DIC method was evaluated to be equal to or better than 10% of image pixel size [15]). Moreover, a relatively small region of interest comprising the FPZ was located around the rotation axis, where reconstruction artefacts caused by the low number of projections are negligible.

The planar X-ray images (radiograms) were taken during specimen loading (between tomography scanning). Acquisitions were taken in 1 s intervals with identical settings of the X-ray tube and geometry. The loading device control and image acquisition were time-synchronized during the overall experimental process. Thus, the force-displacement dependency (or force-time dependency) can be compared with the appropriate X-ray images. Afterwards, mechanical experiment data for beam hardening image correction were collected using a set of aluminium filters with various thicknesses. Projection data were reconstructed using Volex software (Fraunhofer Vision, Germany).
The specimen was placed in the chamber, and its position was centred to align its notch with the axis of the loading fixture. Then, the upper part of the experimental device was attached using a bayonet lock. The upper fixture was slowly lowered to be in contact with the specimen surface. A tomographic measurement of the non-deformed sample was carried out. Then, specimen loading was started, and a load-displacement diagram was recorded and observed in real time. The loading was interrupted at characteristic moments of the observed diagram, and tomographic scanning was performed to capture the three-dimensional situation of the FPZ propagation.

3 Results

3.1 Load-displacement diagram

The load-displacement diagram recorded during the experiment is depicted in figure 5. The measured force suddenly dropped after reaching its maximum value (light blue ring) when the first crack in front of the notch occurred. The quasi-brittle behaviour (slowly decreasing loading force — blue part of the curve) of the material is apparent in the right part of the load-displacement diagram where the loading force asymptotically decreased to zero. Tomographic measurements (green leader lines) were taken in loading states manifested in the graph by short vertical drops of force value that are caused by material relaxation during tomography.

![Image of setup](image-url)

**Figure 4.** Setup used for radiographic and tomographic measurement with the implemented loading device.

**Figure 5.** Load-displacement diagram.
3.2 Planar radiography and 3D crack propagation

Planar X-ray images taken during the loading test allow identification of overall FPZ evolution (see figure 6 illustrating the FPZ shape at different loading displacements). The corresponding loading steps (LS) are depicted in the load-displacement diagram in figure 5. X-ray images are cropped for better FPZ visualization. False colors represent relative material density, where 100 % means the original specimen’s thickness (data were normalized by the signal corresponding to the nominal specimen thickness). Note that it is difficult to distinguish the FPZ from initial material density and sample porosity variations which were first analyzed in radiograms.

Figure 6. Series of planar X-ray images at different loading steps. FPZ evolution is clearly visible from loading step 3. Images are in mm scale; the notch tip has coordinates [0, 0].

The influence of initial porosity and density variations on FPZ identification are suppressed in the differential images as a referential radiogram taken at the beginning contains the same initial porosity and density variations. The actual differential image $R_{\text{diff}}$, with succession number $n$ is calculated as follows:

$$R_{\text{diff}} = 100 \times \left( \frac{R_{\text{eff}} - R_{\text{act}}}{R_{\text{eff}}} \right) \text{[\%]}$$

where $R_{\text{act}}$ is the actual radiogram and $R_{\text{eff}}$ is the referential radiogram. Visibility of the FPZ was significantly improved when comparing actual X-ray images and referential image, see figure 7. The differential X-ray image represents the normalized difference between the actual X-ray image and the referential image. Movement of the specimen’s centre due to loading is taken into account for this calculation. Data range of differential image is at the end inverted ($100 - R_{\text{diff}}$) to emphasize relative density reduction. The influence of material density variations on FPZ identification are suppressed in the differential images. FPZ evolution is visible for all analysed loading steps; however, the FPZ’s 3D spatial distribution cannot be assessed.

The entire reconstructed specimen with support pins is depicted in figure 8 (at left). The volume of interest containing the notch and the newly developed FPZ was selected for better visualization, see figure 8 (at right). It has to be emphasized that the silicate composite contains a number of bubbles which interfere with identification of the FPZ evolution.

The identification of the spatial distribution of the FPZ was carried out by analysis of CT results in four (4) different loading steps (see figure 9). Inspection of the data indicates the specimen’s
Figure 7. Series of planar X-ray radiograms. Color corresponds to the relative density change, 100% means the original density. The FPZ shape is clearly visible from loading step 1.

Figure 8. 3D visualization of the whole specimen (left) and cropped region of interest (right).

Figure 9. 3D visualization of crack evolution during loading test.
steady state until reaching the maximum loading force. The first tomography data were recorded before reaching the maximum loading force (CT_01) as evolution of the FPZ and a consequent decreasing of the loading force is very fast for quasi-brittle materials. A detectable decrease in the density in the FPZ after reaching the maximum loading force (CT_02), with increasing load crack branching, starts in this area (CT_03, CT_04). The fact that crack branching flows through the area with lower material density and higher sample porosity is demonstrated in figure 10.

4 Conclusions

Crack and FPZ evolution, as well as structural changes in the vicinity of the crack, in a cementitious composite specimen were successfully observed using X-ray radiography and X-ray tomography. It was proven that the process zone and crack evolution are closely connected with areas of lower density. The crack grows and branches in directions of lower resistance, i.e. directions with the highest porosity. Also, the shape of the process zone is determined by the local composition of the material. It was also proven that the described methodology provides detailed information about FPZ evolution which cannot be analysed using standard optical methods.

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