Visualization of self-healing materials by X-ray Computed Micro-Tomography at UGCT

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Keywords: X-ray computed micro-tomography, self-healing

ABSTRACT

This work presents recent advancements in X-ray micro-computed tomography (XRMCT) of self-healing materials at Ghent University's Centre for X-ray Tomography (UGCT). Results of XRMCT imaging in a self-healing polymer system are shown to demonstrate the use of XRMCT in self-healing studies. Furthermore, two new XRMCT scanners are presented. The HECTOR scanner was designed for large samples and strongly attenuating samples, and is therefore well suited to study self-healing concrete. The EMCT scanner is well suited for dynamic self-healing experiments in a controlled environment.

1. INTRODUCTION

This work presents recent advancements in X-ray computed micro-tomography of self-healing materials made at Ghent University’s Centre for X-ray Tomography (UGCT). UGCT designs and assembles its own scanners in room-sized bunkers to optimize flexibility and research possibilities. Furthermore, it develops CT reconstruction and analysis software and performs research into both the underlying physics as the advanced application of X-ray computed micro-tomography (XRMCT). XRMCT is a widely used, non-destructive, three-dimensional imaging technique with great potential for studying self-healing materials. As an example, we present results from self-healing polymers. Furthermore, we present two new set-ups, named HECTOR and EMCT, well suited for respectively the study of self-healing concrete and (dynamic) self-healing processes in a controlled environment.
2. X-RAY COMPUTED MICRO-TOMOGRAPHY

X-ray computed tomography can provide a fully three-dimensional image of the interior of an object by reconstructing slices using two-dimensional radiographs of the object, made from a large number of different angles. In each voxel (three-dimensional pixel) of the three-dimensional image the local X-ray attenuation coefficient is obtained, which depends on local densities and effective atomic numbers. As such, it is possible to visualize the internal structure of the sample. A laboratory-based XRMCT set-up consists out of three main parts: an X-ray tube, a rotation stage to mount the sample on and an X-ray detector.

3. SELF-HEALING POLYMERS

As an example of the use of XRCMT for the study of self-healing materials, we show some results from a capsule-based self-healing polymer system. Microcapsules containing healing agents are embedded in an epoxy thermosetting matrix to provide the self-healing property. The microcapsules break upon rupture of the matrix and release the healing agents in the crack, where these react to form a new healed polymer network. Figure 1 shows a CT-scan from a subsample of a Tapered Double Cantilever Beam test sample that was allowed to self-heal after breaking. Single microcapsules can be clearly distinguished to study for example capsule size, shape and spatial distribution. Furthermore, the crack surface can be visualized, allowing to study for example tail formation and delamination of microcapsules at this surface.

![Figure 1: slice through the interior of a self-healing polymer sample (left), 3D rendering of the crack in this sample (right).](image)

4. HECTOR AND EMCT

HECTOR (High Energy CT Optimized for Research) was designed to extend UGCT’s imaging capabilities to larger and/or stronger attenuating objects. Furthermore, there was a need to reduce scanning times for monitoring for example dynamic events. An important component of HECTOR is an X-RAY WorX XWT 240-SE microfocus source. This high power directional tube can generate up to 280 W target power to get a high X-ray flux. The focal spot’s minimum size of 4 μm ensures that the system is still useful for standard micro-CT scanning. A large 40x40 cm² PerkinElmer 1620
CN3 CS flat panel detector allows for a large field-of-view. A rotation stage, specifically chosen to be able to handle heavy loads with extreme precision, was mounted on a vertical stage with a travel of 1m to make helical scanning of long objects (e.g. drill cores) possible [1].

HECTOR’s characteristics make this system specially suited to study concrete drill cores (figure). The system will be used to study self-healing geometries and processes in the framework of the SECEMIN research project.

![Figure 2](image-url)  
**Figure 2**: Reconstruction of a slice of a concrete core with a diameter of 12 cm, scan performed with HECTOR

The EMCT (Environmental Micro CT) scanner was designed to image samples under controlled environmental conditions (temperature, pressure, humidity) or during dynamic experiments (e.g. loading). Therefore, the system is designed having a «rotating X-ray source and detector»-assembly, contrary to conventional XRMCT scanners in which the sample is rotated. Communication, interlocks and power are transmitted over slip ring contacts embedded in the system’s granite base. Therefore, the source-detector combination can continuously rotate around the sample which remains stationary with regard to the outside world. This facilitates connecting external equipment (pumps, loading stages, etc.) to the sample during imaging.

The system has a directional X-ray tube with a minimal spot-size of 5 µm and a maximal accelerator voltage and power of 130 kV and 39 W, respectively. Its CMOS detector with CsI scintillator allows for fast CT scans (up to 300 fps), with a sufficiently small angular interval between successive projections.

![Figure 3](image-url)  
**Figure 3**: The EMCT scanner (left) and HECTOR (right)
The source and detector are mounted on a linear motorised stage to allow changing the magnification. The goniometer on which this stage is mounted has a bore hole of 190 mm diameter through which the samples can be positioned into the beam. The EMCT scanner has a vertical stage with 80 cm travel. This will allow helical scanning of long samples, e.g. drill cores. Using a 3D resolution pattern, we have demonstrated that 5 µm spatial resolution can easily be achieved in the reconstructed CT volume.

5. CONCLUSIONS

XRMCT is a powerful imaging technique with great potential to aid characterization of self-healing materials. With the addition of two new XRMCT scanners, UGCT can now accommodate experiments on self-healing concrete and dynamic experiments.

ACKNOWLEDGEMENTS

The Agency for Promotion of Innovation by Science and Technology in Flanders, Belgium (IWT) is acknowledged for the PhD grant to Tom Bultreys. The work presented is part of the Strategic Initiative Materials (SIM) project, supported by the IWT.

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