CHARACTERIZATION OF DISTRIBUTED DAMAGE AND SELF-HEALING IN CEMENTITIOUS MATERIALS BASED ON TIME-DEPENDENT 3-D X-RAY COMPUTED MICROTOMOGRAPHY (MICRO-CT)

S. Fan[1] and **M. Li** [1]

[1]Department of Civil & Environmental Engineering, University of Houston, 4800 Calhoun Road, Houston, TX 77204 – e-mail:sfan@uh.edu; moli@uh.edu.

Keywords: x-ray computed microtomography, nondestructive, damage, self-healing, engineered cementitious composites,

ABSTRACT

Concrete cracking is inevitable, and can be the result of one or a combination of factors such as dry shrinkage, thermal contraction, fatigue, and embedded steel corrosion. The presence of cracks leads to further deterioration, service life reduction of concrete infrastructure, and frequent maintenance and repairs. These challenges can be potentially addressed with innovative self-healing cementitious materials, which can autogenously regain material transport properties as well as mechanical characteristics after the damage self-healing process.

For the development of self-healing cementitious materials, it is crucial to precisely characterize the extent and quality of self-healing due to a variety of factors. X-ray computed microtomography (Micro-CT) was adopted in this study to derive three-dimensional tomographic data of micro-cracks before and after healing in engineered cementitious composite (ECC) materials. This method is a non-destructive visualizing technique that allows digitalization and monitoring of the interior characteristics of solid objects. ECC specimens were pre-damaged under bending to form multiple micro-cracks, and then exposed to wet-dry cycles to allow potential self-healing to occur. Micro-CT was then employed to build 3-D tomography models of the samples. The 3-D microcrack geometry, width and area were quantified. The extent of self-healing was then determined. The results were further combined with scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) to characterize crystalline and chemical properties of the self-healing products.

This study showed that Micro-CT is a suitable advanced technique to directly quantify self-healing potential in solid materials. The Micro-CT results revealed that self-healing extent of ECC is strongly influenced by crack width. For a bending crack with surface crack width of 30 μm, 55.3% of the crack volume was healed after 5 wet-dry cycles. For a bending crack with surface crack width of 100 μm , only 7% of the crack volume was healed after 5 wet-dry cycles. Hence, controlling microcrack width to under 30 μm is a necessary condition for achieving early, robust self-healing in ECC.

1. INTRODUCTION

Self-healing cementitious materials, which can autogenously recover transport properties as well as mechanical capacity after cracking, can greatly extend service life of civil infrastructure with minimum repairs. Engineered cementitious composites, or ECC, shows great potential of developing robust self-healing cementitious materials because of its unique and inherent cracking control capacity [1]. ECC features a strain-hardening behavior accompanied by multiple micro-cracking, which is in contrast with the localized cracking or fracture in conventional concrete materials. As a result, the micro-crack width in ECC during strain-hardening is independent of steel reinforcement ratio, applied deformation and member geometry, offering one of the most important prerequisites for self-healing. Self-healing in ECC has been studied in terms of recovery of transport properties and tensile stress-strain relation under various environmental exposure conditions [2-5].

Self-healing characterization techniques for ECC and concrete materials include: water permeability and signal transmission tests to characterize transport properties, ultrasonic echoing and dynamic modulus measurement to characterize both transport and mechanical properties, as well as destructive tests such as bending, compressive, uniaxial tension and stiffness measurements. Additionally, analysis techniques of self-healing products include SEM and AEM (analytical electron microscopy) imaging, EDX and XRD (X-ray diffraction) chemical analysis, and nano-indentation. These methods have been effective to characterize self-healing in cementitious materials at the bulk composite material level, or to analyze self-healing products present at the sample surface. However, until now collecting full data on the 3-D microstructure of cementitious materials is still a difficult task. This paper focuses on the nondestructive and 3-D characterization of distributed damage and self-healing in ECC through Micro-CT. For the first time, the self-healing extent of 3-D bending cracks within ECC was directly and accurately quantified.

2. MATERIALS AND SPECIMEN PREPARATION

ECC specimens were prepared using the mixture proportion in Table 1. The fresh ECC mixture was cast into a series of coupon specimens with dimensions of 300× 76.2 ×12.5 mm. The specimens were covered with plastic sheets and cured in laboratory air with a temperature of 20±1°C and relative humidity of 45%±5. At the age of 45 days, the specimens were tested under four-point bending until failure. A number of microcracks with width ranging from 20 to 120μm were generated at the tensile side of the specimen. After unloading, the crack patterns of the specimens were examined under an optical microscope. Two 10×10×11 mm cubic samples with different average crack width were selected and cut from the tensile side of coupon specimens. Sample A contains 4 similar-size microcracks with average surface crack width of 30μm; Sample B contains 3 similar-size microcracks with average surface crack width of 100μm. The two samples were then exposed to 5 wet-dry cycles to allow potential self-healing to occur. For each cycle, the samples were first submersed into water at 20°C for 24h, and then naturally dried in ambient air at 20±1°C and 45%±5 RH for 24h. Micro-CT was conducted on the samples before and after self-healing to build 3-D tomography models.

Table 1: ECC mixture proportion

Mix	W/(C+F)	Cement	Sand	Fly ash	Superplasticizer	Fiber
		Kg/m^3	Kg/m^3	Kg/m^3	Kg/m^3	Vol-%
ECC	0.21	461	370	1015	2.8	2

3. X-RAY COMPUTED MICROTOMOGRAPHY CHARACTERIZATION RESULTS

The principle of X-ray computed tomography is based on the 3D computed reconstruction of a sample from 2D projections acquired at different angles around its axis of rotation. Figure 1(a) shows one of the raw 2D images of sample B before self-healing, which was converted into binary images and then the final images of ROI (region of interest) after the air pores were removed. Figure 2 shows the 3D image of sample B after reconstruction that combines all the 2D images.

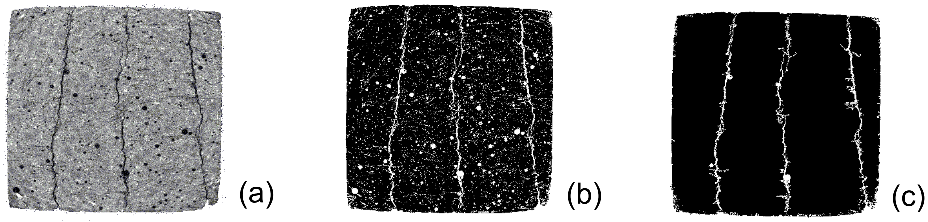

Figure 1: 2D images of slice 1652 for sample B before self-healing: (a) raw images built from tomography data set; (b) binary image; (c) final processed image of ROI

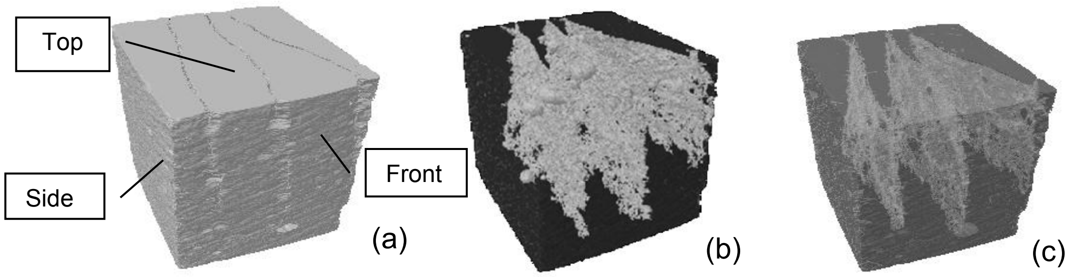

Figure 2: 3D images model for sample B: (a) original reconstructed 3D image before self-healing; (b) final processed image of ROI before self-healing ; (c) final processed image of ROI after self-healing

Sample A has an average surface crack width of 30μm; its average crack width along the crack depth is 15μm. After 5 water/air cycles the total crack volume decreased by 55.3%. Sample B has an average surface crack width of 100μm; its average crack width along the crack depth is 55μm. Only 7.5% of the total crack volume was healed, which is not as significant as sample A. Figure 3 shows the crack area and normalized crack width as a function of crack depth before and after self-healing. It was obvious that sample B, due to its larger initial crack width, has much less extent of self-healing. Figure 4 shows the extent of self-healing along the crack depth in two single microcracks in sample A.

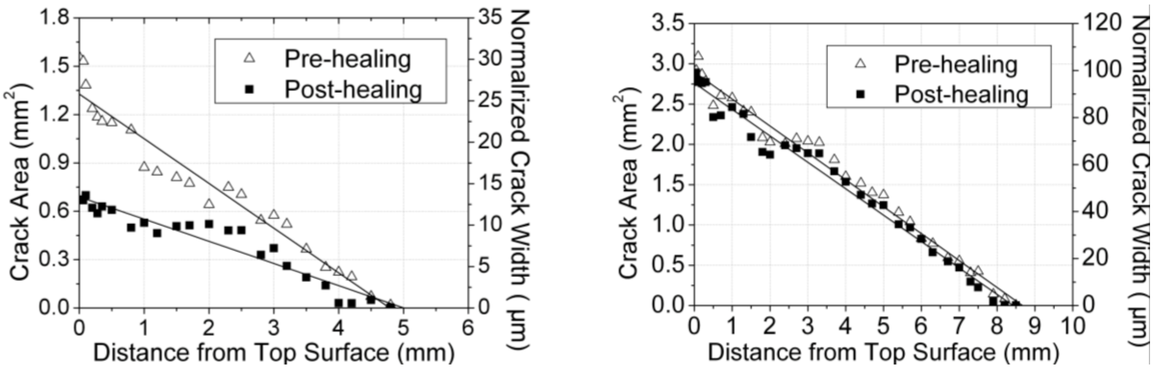

Figure 3: Self-healing extent of micro-cracks within sample A (left) and B (right)

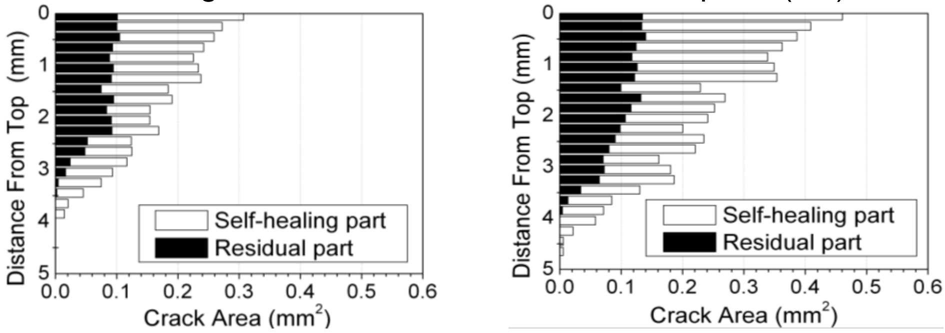

Figure 4: Crack area of sample A before and after self-healing: crack #1 (left); crack #2 (right)

4. CONCLUSIONS

Micro-CT is an effective non-destructive technique to directly characterize self-healing extent in three dimensions. This is extremely important when the cracks do not have a uniform geometry along their depth, such as bending cracks, so that self-healing characterization from the surface is not sufficient. Furthermore, compared to other techniques that provide bulk information of the self-healing extent, Micro-CT can offer direct measurement of each crack. Based on the Micro-CT results, we found that the extent of self-healing strongly depends on the initial crack width, which must be controlled to be very tight for achieving early and robust self-healing. Self-healing extent also depends on crack depth. The microcracks close to the surface tend to heal sooner because it takes time for water or carbon dioxide to transport through the micro-cracks to react with unhydrated cement or calcium hydrate to form self-healing products (i.e. C-S-H and calcium carbonate). The crystalline and chemical properties of the self-healing products were studied with SEM and EDS, and will be reported in a separate paper.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge Dr. Roberto Fajardo and Dr. James Schmitz at the Micro-CT core facility at the University of Texas Health Science Center.

REFERENCES

[1] V.C. Li, On engineered cementitious composites (ECC) — a review of the materials and its applications, Journal of Advanced Concrete Technology, 1, (2003) 215–230.

[2] Y. Yang, M.D. Lepech, E. Yang and V.C. Li, Autogenous healing of engineered cementitious composites under wet-dry cycles, Cement and Concrete Research, 39, (2009) 382-390.

[3] M. Li and V.C. Li, Cracking and healing of engineered cementitious composites under choloride environment, ACI Materials Journal, 108(3), (2011) 333-340.

[4] L.L. Kan, H.S. Shi, A.R. Sakulich and V.C. Li, Self-healing characterization of engineered cementitious composite materials, ACI Materials Journal, 107(6), (2010) 617-624.

[5] E.N. Herbert and V.C. Li, "Self-healing of engineered cementitious composites in the natural environment", in Proc., Sixth Int'l Workshop on High Performance Fiber Reinforced Cement Composites (HPFRCC6), 2011, pp. 148-155.