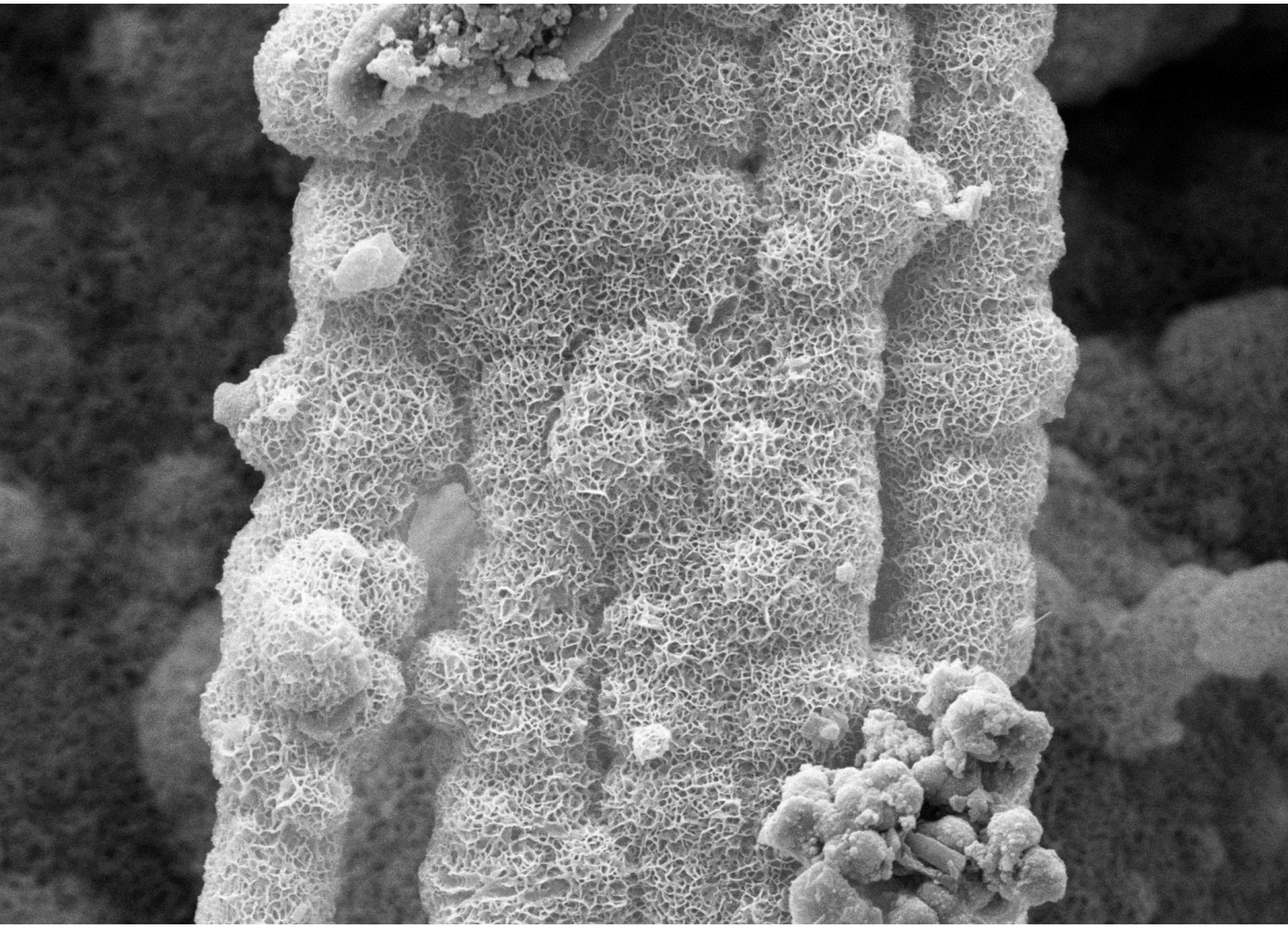
Technology Note



Concrete Crack Self-healing Materials
Micro Structure Investigation

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Introduction

Since the nineteenth century, concrete has become the most widely used material in human history. Almost all civil engineering infrastructures are completely or partly built with concrete. This increasing use of concrete is due to the invention of modern cements. Although there have been many improvements in the materials used in cement, cracks in concrete due to loading and environmental conditions are a major source of reduced durability and increased financial losses. To mitigate this problem, a research project has started at the University of Cambridge to improve the self-healing capacity of cementitious materials in order to create self-healing concrete.

The concept of self-healing in concrete is inspired by the biological environment where living organisms can repair themselves when they are damaged within certain limits [1]. In this research, potential minerals (magnesia, bentonite clay, quicklime) were used in the cement, which have the property of expansion when any crack forms [2]. This expansion of minerals fills the crack and bridges it over time.

Figure 1 *Self-healed sample*

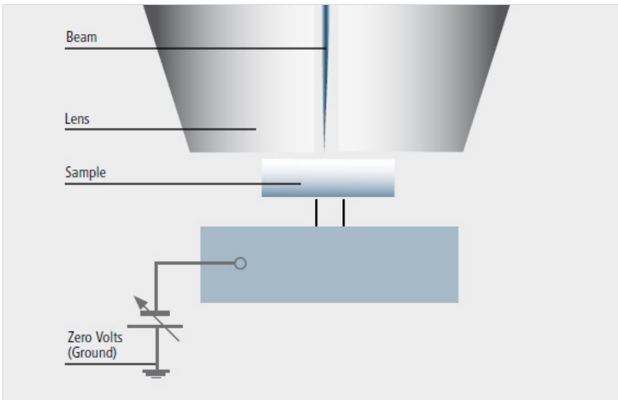

Figure 2 *SEM technique, the interaction of the electron beam with the sample generates secondary electrons (SE) which are collected by electron detectors.*

Part of the research project involved identifying the self-healing materials forming and bridging the crack. The material microstructure was investigated using EVO LS 15 Scanning Electron Microscopy (SEM), which can capture high definition topographical images of the self-healing bridges. This application note demonstrates the use of the EVO SEM for investigating the microstructure of crack bridging self-healing materials.

Instrumentation

EVO LS 15 was used to obtain high definition secondary electron (SE) images of the crack bridging self-healing materials.

The self-healed samples were imaged in high vacuum at 12 keV beam energy and short working distance using OptiBeam resolution mode. The SEM technique is shown in Figure 2. The electron beam is focused onto the sample section and secondary electrons are detected with the secondary electron detector. This detector provides images rich in topographic detail based on the electron intensity received by the detector.

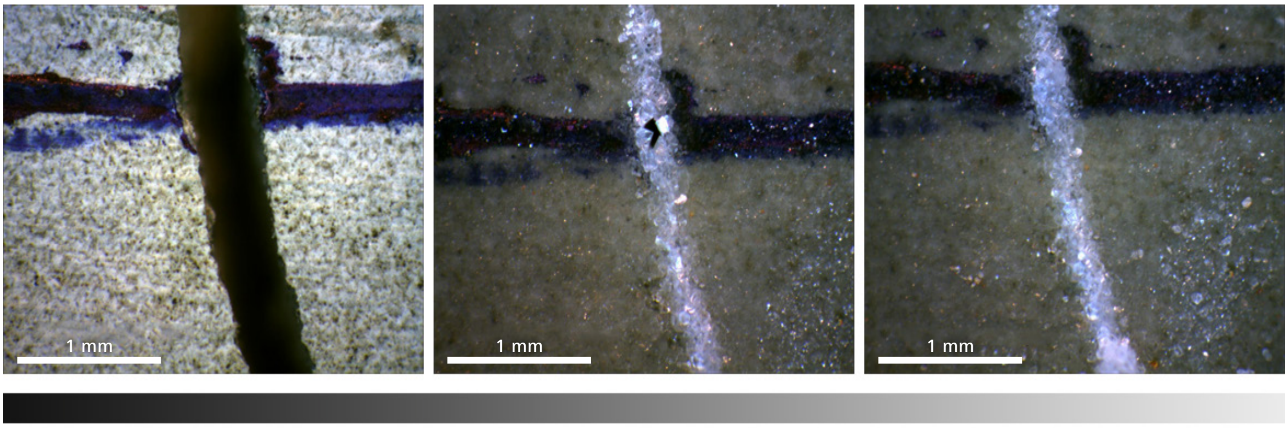

Figure 3 *Image of crack healing in the cementitious materials monitored over time using a digital light microscope.*

Experimental method

Crack healing in the cement matrix was monitored using a digital light microscope as a function of time in order to visualise the healing process. (Fig. 3).

Small samples were collected from the crack self-healed zone whilst taking care not to disturb the top surface of the self-healing zone. Experimental specimens were then soaked in acetone immediately after collection and left for three days to stop further reaction and remove any chemically bound water. Afterwards samples were dried in an oven at 60 °C for three days, attached to an SEM stub and gold coated to aid collection of topographical detail in the SEM. SEM images were obtained on the specimen surface in order to investigate the micro structure of the self-healed bridges.

All images were obtained in high vacuum with an electron beam energy of 12 keV at short working distance and utilising OptiBeam resolution mode. The EVO secondary electron detector (SE) was used to obtain high resolution topographical images of the surfaces to reveal the microstructure.

Micro structure image investigation

The SEM images in Figures 4 and 5 shows the micro structure of the self-healing materials. It can be seen from the images that the bridge structure formed in the crack is composed of a mix of minerals and these healing materials can be identified based on their structural pattern and formation style.

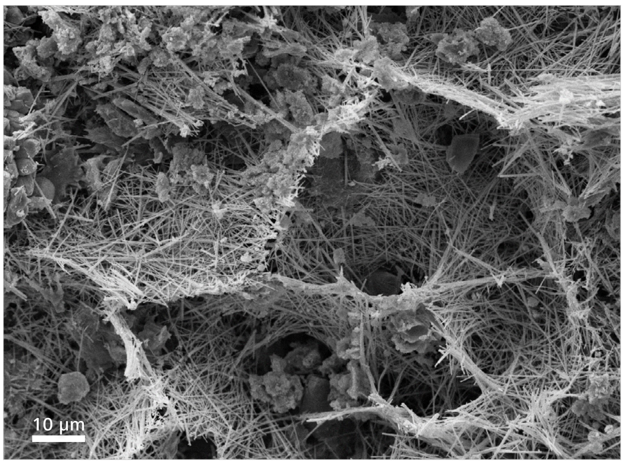

Figure 4 *Self-healing minerals expansion and crack bridging network imaged with ZEISS EVO SE detector with a beam energy of 12 KeV confirms the formation of Ettringite scaffolding structures.*

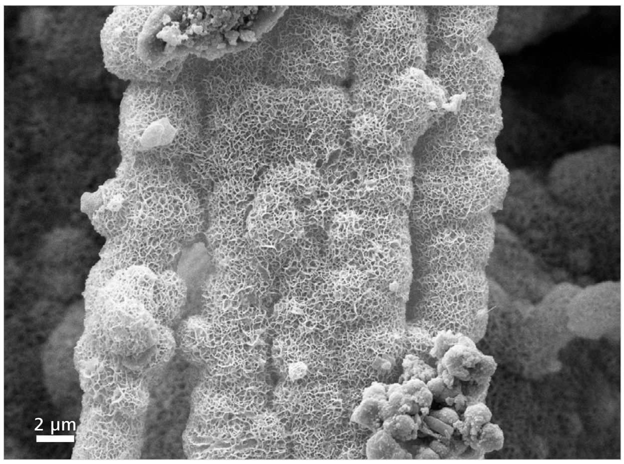

Figure 5 *Self-healing minerals expansion and crack bridging network imaged with ZEISS EVO SE detector with a beam energy of 12 KeV shows flower-like hydromagnesite structures had formed.*

The most commonly used cement in concrete is Portland Cement (PC) which has fast hydrating and hardening properties. Expansive minerals (magnesia, bentonite, quicklime) remain mostly unhydrated within the quick hydrating PC matrix. However, when a crack forms, the expansive minerals become available to react and bridge the cracks.

The expansive minerals that have been used in the different cement mixes for self-healing concrete formed different kinds of healing compounds based on their mix proportions. This was due to the difference in functionality of those minerals in the self-healing process. Magnesia serves expansion and bonding, bentonite serves expansion and scaffolding for crack bridging expanded minerals, and quicklime serves the functionality of crystallisation, which enhance the healing performance [2].

The SEM image of the healing materials presented in Figure 4 confirms the formation of Ettringite scaffolding structures. Other healing compounds such as calcite, portlandite, magnesite expanded within this ettringite network, which filled and bridged the cracks.

Higher magnification imaging of a sample containing highly reactive magnesia shown in Figure 5 confirms that flower-like hydromagnesite structures had formed. This had effectively expanded within the crack, sealing and healing the crack.

Conclusion

SEM topographic imaging of self-healing concrete materials provides microstructural information of the materials that had formed during healing process.

Using these images it is possible to identify the composition and mix of self-healing materials based on their structural pattern and formation style, including scaffolding structures, flower-like structures and other bridging and filling structures produced by different expansive materials during the healing process.

These findings were further validated by other microstructural investigations, which were coherent with the SEM image findings.

References:

[1] Scott R. White, benjamin J. Blaixzik, Sharlotte L.B. Kramer, Solar C. Olugebefola, J.S.M. and N.R.S., 2011. Self-healing Polymers and Composites. American Scientist, (October). Available at: www.americanscientist.org

[2] T. Qureshi, A. Al-Tabbaa, The effect of magnesia on the self-healing performance of Portland cement with increased curing time, AMS, 14 Proceedings of the Int. Conference on Ageing of Materials & Structures, Delft 26 – 28 May 2014, The Netherlands, (2014), 639 – 642

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