Efficiency of self-healing agents for cementitious materials characterized by NDT

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Abstract. The combination of the different material components of concrete (cementitious matrix, aggregates) is beneficial for many applications and constructions. Besides all the positive properties, concrete structures are subjected to cracking or spalling when in service. When cracks exceed a certain width, the increased permeability leads to moisture transport which can finally reduce the service life. To overcome this problem, self-healing concrete is being developed. Various self-healing mechanisms have already been tested \cite{1,2} and will further be evaluated. To commercialize the technique to achieve market acceptance a proof of efficiency of the healing process is required. After first experiments using non-destructive testing methods, enhanced and more sophisticated techniques demonstrate the reliability and reproducibility of the methods. Crack-controlled three-point bending experiments, monitored by acoustic emission technique, have been conducted. Ultrasonic transmission measurements revealed corresponding results for a quantification of the capillary flow in terms of polymeric healing agents in concrete. The determination of the elastic moduli of specimens by vibration analysis reveal a regain of mechanical properties after healing. Further investigations will show, if these techniques are also applicable for test specimens on a large scale or even in field.

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Keywords: Acoustic emission, ultrasonic transmission, vibration analysis, self-healing concrete

1. Introduction

Self-healing is a well-known phenomenon in cement-based materials. As small cracks have been observed by autogenously healing with time the crack width should not exceed a prescribed limit. As a consequence modified concrete is going to be developed to regain more resistance – for example against ingress of aggressive substance to avoid corrosion. In the 1990’s, when self-healing properties were already implemented for polymeric materials and thermoplastic systems, Dry \cite{3} started to work on self-healing concrete. Self-healing can be classified into autogenic and autonomic healing processes. With the objective to enhance the recovery against environmental and mechanical actions in contrast to natural healing or sealing, material components as superabsorbent polymers (SAPs), bacteria and encapsulated liquid healing agents are investigated within an international research project funded by the European Commission. To verify crack sealing or crack healing of these
materials experimental techniques are necessary. While sealing effects are mostly examined by permeability tests [4, 5], mechanical testing is the preferred choice to verify regain in strength for self-healing of cracks [6, 7]. In order to characterize the effects of autogenic/autonomic healing mechanisms and to improve the development of self-healing concrete with as little interferences as possible, non-destructive testing (NDT) methods are applied. In contrast to traditional testing methods as mentioned before NDT methods additionally have the potential to monitor and evaluate the performance in-situ in large-scale or field experiments. Preliminary studies showed that techniques like ultrasound [8], resonance frequency analysis [9] and acoustic emission analysis [10, 11, 12] are suitable methods for evaluation.

Due to the wide range of different healing agents and its specific field of application this article presents selected results of small-scale experiments based on previous results [13] as part of preliminary studies for upcoming large-scale or in-field tests.

2. Test setup

2.1 Specimens and healing agents

For the studies presented in this paper reinforced concrete specimens with a dimension of 150x150x550 mm have been used. The concrete composition and the average cube strength (150x150x150 mm) is given in Table 1.

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Composition kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement CEM I 42.5N</td>
<td>300</td>
</tr>
<tr>
<td>Water</td>
<td>150</td>
</tr>
<tr>
<td>Sand 0/2</td>
<td>686</td>
</tr>
<tr>
<td>Gravel 4/8</td>
<td>502</td>
</tr>
<tr>
<td>Gravel 8/16</td>
<td>809</td>
</tr>
<tr>
<td>Mean compressive strength (28 days)</td>
<td>51.0 MPa</td>
</tr>
</tbody>
</table>

A commercial polyurethane based healing agent (MEYCO MP 355 1K) encapsulated in glass capillaries was placed in the middle section of the mould (Fig. 2). The prepolymer of polyurethane is developed for injections to stop flowing water in cracks by becoming foam. To shorten the reaction time additional capillaries with a mixture of accelerator and water were added. Fluorescent pigments simplify the detection of the surface covered with the healing agent which provides information about its dispersal characteristics used for comparisons with NDT results. Furthermore, concrete beams without glass capsules were casted for manual filling (with a syringe) with a super low viscosity polyurethane (SLV) as seen in Fig. 1. For the fixing of the capsules thin steel wires were used (Fig. 2, right).
2.2 Loading

To create controlled cracks, specimens were loaded in a crack-width-controlled 3-point bending test with a speed of 0.06 mm/min up to 0.9-1.0 mm. After unloading the beam the crack shrinks to a width of around 0.35-0.45 mm. A predefined notch guaranteed to fracture the beam in the middle section. In consequence, capsules break and the healing agent can flow and polymerize. After 7 days of hardening at 20°C and 65% RH, specimens were reloaded using the same setup.

2.3 NDT setup

Acoustic emission (AE) techniques [14] were applied during bending to provide information about the cracking behaviour, capsule breakage and healing efficiency. In total 15 ultrasonic p-wave transducers (V103 by Olympus, Panametrics) were coupled at the concrete surface surrounding the middle section for AE as well as an s-wave transmitter and receiver (V150 by Olympus, Panametrics) at the outer sides for direct ultrasonic transmission measurements (Fig. 2).

Fig. 2. Drawing representing a concrete beam with embedded self-healing agents (4 pairs of capillaries)

Preliminary tests with pencil-lead breakages provided information about wave speed and triggering parameters. In addition it ensures a correct sensor placement for localization of AE in the significant area to be examined.
For the monitoring part ultrasound pulses were transmitted every second to analyse the fracture process and the subsequent healing progress in a direct comparison. To reduce side effects as sensor coupling the s-wave sensors remained attached throughout the investigation.

3. Results

3.1 Acoustic emission analysis

In Fig. 3 the AE activity and the corresponding loading curve of two concrete beams during reloading is shown. The specimen B1 (Fig. 3, top) is healed manually as seen in Fig. 1. Therefore a polymer with a super low viscosity (SLV) was filled into the crack with a syringe. The concrete beam B2 (Fig. 3, bottom) contained 2 pairs of capillaries filled with MEYCO. By comparison of the maximum peak of the loading curves it is noticeable that the regain in strength of B1 is higher than for B2. A further characteristic point is the decay curve until the following minimum point (Fig. 3, marked with a red arrow). The subsequent linear slope is related to a loading of the reinforcement bars and will not be taken into consideration.

The cross-section view of the crack flanks after splitting (Fig 4) reveals a correlation between covered surface with polyurethane and maximum load regain. The AE activity allows...
characterizing the polymers properties (e.g. stiffness, quality of bonding) during failure. A particular focus of the activity range is from the beginning until the first minimum point of each loading curve. While for B1 most events occur during load increase within the observation range until a crack width of 0.37 mm (measured by LVDT), the encapsulated “MEYCO” healing agent causes a constantly higher AE activity until 0.2 mm (LVDT). The reason for this result is explained in the brittle behavior of the MEYCO polymer. In contrast, the SLV polymer features a good strain capability and is applicable for dynamic and cyclic loading [15]. A localization of the recorded AE events of B2 until 0.2 mm crack width returns a correlation with the polymer covered area (Fig. 5). This confirms that the parameter based AE activity (Fig. 3) is related to the failure of the polymer.

![Fig. 4. Cross-section view after splitting the concrete beams. Left: B1 - manually healed with SLV polymer. Right: B2 - encapsulated capillaries with MEYCO polymer](image)

![Fig. 5. Localization of AE events of B2 until crack width 0.2 mm (LVDT). Top: AE events projected into cross-section plane. Bottom: AE events seen in longitudinal (length) plane](image)
3.2 Ultrasonic transmission measurements

Ultrasonic signals transmitted and received by piezoelectric broadband sensors in transmission technique allow extracting characteristic properties of the examined material. Real-time monitoring as an enhanced testing method can provide information of material changes like the hardening of fresh concrete [16, 17, 18] in-situ.

The applied FreshCon system (developed by Smartmote) depends on the determination of the onset time of the transmitted compression and shear wave (p-wave, s-wave). However, since the full waveform of each signal is recorded and saved by the FreshCon-software a detailed signal analysis is possible. These signal based parameters are e.g. maximum/average amplitude, amplitude of single peaks or squared amplitude. Additionally, it enables further evaluation methods, such as coda wave interferometry. In this study approaches of monitoring the fracture process, shrinkage effects due to relaxation and the hardening of the polyurethane based healing agents have been investigated by focusing on the s-wave amplitude. Preliminary tests revealed that the amplitude is much more sensitive and more reliable than the onset time. The setup is described in 2.3.

In Fig. 4, the behaviour of the s-wave amplitude is shown as function of time. During loading the amplitude decreases with increasing crack width. Despite continuous crack opening, after ~5 minutes loading the minimum amplitude is reached. To study the effect of the relaxation and storing conditions (baseline measurement) the concrete has been brought into a climate chamber and the monitoring resumed for ~100 h. Finally SLV polyurethane was filled into the crack (similar procedure as seen in Fig. 1, right) and the amplitude increases rapidly with hardening. The early peak followed by a short drop (~100 h) is remained unclear and demonstrates initial inconsistencies of this evaluation approach.

Fig. 5. Evolution of the s-wave amplitude during loading (top), relaxation (until ~100 h, bottom) and healing (~100 h - ~114 h, bottom)
4. Conclusion and outlook

In this study, self-healing concrete beams with two different polyurethane based precursors have been analysed. Therefore, AE testing and long-term ultrasonic transmission measurements were applied. AE analysis techniques are able to monitor and distinguish between fracture processes of different healing materials. Time-dependent ruptures related to polymer failures allow to characterize the debonding of healed crack flanks during a 3-point bending test. Using the same test setup the AE activity (counting and energy) revealed a distinction in the brittleness of the polymers. A localization of the AE events confirmed the correlation to the polymer’s failure. In contrast, the test specimen with manually filled SLV (B1), implying a more flexible behaviour, causes less AE events with less energy by featuring a higher regain in strength.

Long-term monitoring with transmitted ultrasonic pulses is a useful method to trace the bending as well as the healing process. While baseline measurements reveal almost a monotonous s-wave amplitude, increasing amplitudes indicate a curing process causing a rebond of the crack flanks. Discontinuities in the curve progression are still unresolved questions and demand further investigations. With respect to applications in a practical way (e.g. one-sided coupling, less sensors deployed) ongoing ultrasonic measurements are supposed to detect correlations between waveform parameters and the polymer-covered crack surface. Monitoring the curing time will also be a relevant target of investigations for polyurethane based healing methods in future.

The aim is to apply these NDT techniques to larger self-healing test specimens (with polymers, SAPs, bacteria) like slabs or columns.

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