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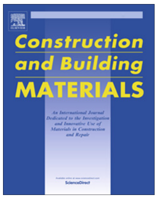

Review

From straw in bricks to modern use of microfibers in cementitious composites for improved autogenous healing – A review

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HIGHLIGHTS

- This review gives an overview of strain-hardening cementitious materials.
- Biomimicry and cementitious materials are linked with each other.
- Improvement of autogenous healing in cementitious materials is discussed.

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ABSTRACT

Cracks in concrete are inevitable and for durability reasons, the cracks should be repaired. Concrete has the intrinsic property to heal itself. But, the passive form of autogenous healing plays only an inferior role for a complete repair of a cementitious material. The main cause is that only cracks of limited width may heal completely. For that reason, microfibers are added to the mixture, as they cause the formation of multiple small cracks. In this way, a ductile material is designed with the property to heal itself efficiently. This paper will overview the different fiber reinforced cementitious composites of the last decade, the link with autogenous healing, results from the literature and future prospects.

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Nomenclature

Chemical abbreviations

Ca^{2+}	calcium ion
$CaCO_3$	calcium carbonate
$Ca(OH)_2$	calcium hydroxide
C–S–H	calcium silicate hydrates
CO_2	carbon dioxide
CO_3^{2-}	carbonate ion
HCO^{3-}	hydrogen carbonate ion
H_2O	water
OH^-	hydroxide ion

Abbreviations

ECC	engineered cementitious composites
FRC	fiber reinforced concrete
FRCC	fiber-reinforced cementitious composites
HTPP	high tenacity polypropylene
HPFRCC	high performance fiber reinforced cementitious composites
MFCFRCC	multiple fine cracking fiber reinforced cementitious composites
PE	polyethylene
PP	polypropylene
PVA	polyvinyl alcohol
SAP	superabsorbent polymer
SHCC	strain hardening cementitious composites
TRC	textile reinforced concrete

1. Introduction

Concrete is a material which can cope with high compressive stresses, but has a low tensile strength. Adding reinforcements increases the strength in tension but the composite will crack nevertheless. Cracking in plain concrete is thus inevitable. It can be the result of one or a combination of factors such as drying shrinkage, thermal contraction, restraints, differential settlement, and applied loads. Cracks are aesthetically unwanted and they will cause durability issues as they form a pathway for intruding potentially harmful substances. After crack formation and water intrusion, the pH will drop in the vicinity of reinforcements, leading to steel corrosion and possible structural declination. The ingress of chlorides will accelerate corrosion by de-passivating the protective film around reinforcements. Intruding carbon dioxide will react with the calcium hydroxide $Ca(OH)_2$ in the pore fluid causing a decrease of the pH and thus de-passivation and increased corrosion. The ingress of sulfates can result in the formation of ettringite, a subsequent volume expansion and a damaged microstructure. All these deteriorating processes need to be stopped before it is too late.

The amount of cracking can be controlled after taking the causes into account and repairs can be applied to seal the cracks from intrusion. But these repairs are time-consuming and costly. It would therefore be beneficial if the material would heal on its own. The self-healing concrete should hereby provide a complete or partial regain of the mechanical properties after crack formation. This happens in situ, meaning that no interaction has to be undertaken like manual repair. This would improve the reliability and the lifetime of structures.

Autogenous healing, which will be discussed later-on, can only close small cracks. A way to obtain these small cracks in concrete is the use of microfibers. In this paper, the history of fiber-reinforced cementitious composites will be addressed first. Different advantages and disadvantages of using different fibers in cementitious materials will be covered like the effects on degradation, steel corrosion and crack width restriction. The second part will be a close-up of autogenous healing and the needs for attaining a complete healing of a cementitious composite. The third part will focus on the basic knowledge on the optimization of self-healing with the addition of microfibers. The future of this kind of self-healing cementitious materials with fibers will be discussed at the end.

2. From straw in bricks to modern use of microfibers in cementitious composites

2.1. Microfibers through history

Fiber-reinforced composites are used frequently nowadays and its properties have been used for a long time. One of the first written references to fiber reinforced composites can be found in the Biblical book Exodus [1]:

> "Pharao praecepit ergo in die illo praefectis operum et exactoribus populi dicens nequaquam ultra dabitis paleas populo ad conficiendos lateres sicut prius sed ipsi vadant et colligant stipulam. – *Exodus* ***5*** *(6–7)*"
>
> "That same day Pharaoh gave the order to the slave drivers and overseers in charge of the people that they are no longer to supply the people with straw for making bricks and that the people should go and gather their own straw. – *Exodus* ***5*** *(6–7)*"

In ancient times the Egyptians, Sumerians, Babylonians and other civilizations used straw or horse hair to reinforce clay bricks. In this way, the bricks were stronger and were more durable in time. In the case of the Babylonians, this reinforcement could be beneficial in view of the liability of the constructor. Think about the Code of Hammurabi, a Babylonian law code, in which the builder is responsible for good practice and would lose his life if the owner of the house was killed due to poor construction. The use of reinforced materials was therefore imposed and the composites are still used today.

In 1963, fiber reinforced concrete found its way to the scientific community [2,3] and since then, the material has been intensively studied. Review papers on fiber reinforced cement-based composites can be found in [4] and [5]. Generally, fiber reinforced concrete is a material containing dispersed randomly oriented fibers. There are several materials which can be used in cementitious materials: natural fibers (e.g. akwara, bamboo, cellulose fibers, coconut husk, elephant grass, flax, hemp, jute, malva, musamba, plantain, sisal, sugar cane bagasse, water-reed, wood), glass fibers, carbon fibers, metal fibers (e.g. alumina, steel), and synthetic fibers (e.g. acrylic, aramid (Kevlar), nylon, polyester, polyethylene, polypropylene, polyvinyl alcohol) [1–34]. Also asbestos was used, but that material is has been banned due to human health issues.

2.2. Glass, steel and natural fibers: degradation in an alkaline environment and corrosion

One of the main concerns of the use of non-cementitious materials in a cementitious matrix is the (change in) alkaline environment. The strength of the composite may decrease and in case of the fibers, the bond with the matrix can change due to chemical and physical interactions, or the fibers may degrade. These interactions are due to the alkaline pore fluid (pH > 13) combined with the intrusion of potentially harmful substances through a crack. The alkaline environment poses a threat for glass fibers [20,21,35] and natural fibers [36–41], but almost none for synthetic fibers. Si–O–Si bonds are destroyed in the glass network due to the alkaline environment [35]. By making the glass fibers alkali resistant, this can be overcome [17,22,42]. Also natural fibers can be surface-treated [26,43]. On the other hand, the matrix can be modified too, to lower the alkalinity [18,44] and thus the degradation in this alkaline environment. The degradation due to intrusion by substances changing the alkalinity occurs mainly with steel fibers due to corrosion [45]. Generally, all degradation processes will lead to a decrease in strength of the composite material.

A good review on cementitious building materials reinforced with vegetable fibers can be found in [8,46]. In natural fibers, the molecular chains of hemicellulose degrade due to the alkaline environment. There are three main processes involving degradation in an alkaline environment [47]. The first one is peeling off due to the reaction of alkaline cations with hydroxide groups OH^- of cellulose. However, due to the high degree of polymerization and the slow kinetics, this process is negligible. The second main process is called alkaline hydrolysis in which the molecular chains of hemicellulose are broken, resulting in a loss of strength [6]. The third one is the petrification of a natural fiber. In this process, the hollow part of the natural fiber, the lumen, is hardened with cement products [28]. By lowering the amount of alkali-sensitive hemicellulose by chemical or physical methods, the sensitivity decreases. As some natural fibers like flax or hemp have excellent strength properties, the natural fibers could be a low-cost solution to the demanding quest to obtain an alternative for the synthetic fibers [6].

The problem, however, remains the alkali-sensitivity. Further investigations must show the possibility of natural fiber reinforced cementitious materials to obtain a strain hardening effect. The durability is hereby an important factor to be considered. Chemically treating these fibers may give a solution of the degradation of natural fibers in an alkaline environment. In the end, this may lead to a greener composite for autogenous healing. Unpublished results show that mercerization with 2 m% concentration sodium hydroxide (NaOH) resulted in optimal multiple cracking of mixtures containing flax and hemp [48]. A ductile strain of 1% was feasible.

2.3. Synergetic interaction between fiber and cementitious matrix

One of the reasons why fibers found their way in the concrete technology is the replacement of reinforcements and the control in cracking behavior. As concrete is a brittle material, reinforcements are placed when casting the concrete to take up the tensile loads. The reinforcements are located in distinctive places. Fibers on the other hand, are evenly distributed in the matrix. They are not able to withstand large tensile stresses, but due to their uniform distribution, they can control the cracking behavior.

Before going into detail about the effects of synthetic fibers in cementitious materials, the interactions need to be understood. The properties of the cementitious matrix, the fiber and the transition zone between both are important. The three combined affect the properties of the cementitious composite. There are also three main factors regulating the bond between fiber and matrix [1]. These are (1) friction, (2) anchorage due to deformations and (3) physical and chemical adhesion. The bond can be measured with a simple pull-out test [49–51] or by investigating the protruding fiber length from a tensile fracture surface of the cementitious composite [52]. In the latter, the bond is linked to the protruding length as a long embedment will cause rupture of the fibers if the bond is too strong. In this way, the critical length can be determined.

When a fiber is pulled out, the bond with the matrix is theoretically lost, and pure friction takes over. However, there can be slip softening and slip hardening. The latter happens due to blockage in the fiber tunnel when a fiber is pulled out. The retaining forces of the fibers are dependent on the embedded length in the matrix. This is the length of a fiber at a side of a crack which is surrounded by plain material. If the embedded length is too small, the fiber will be pulled out without any slip hardening.

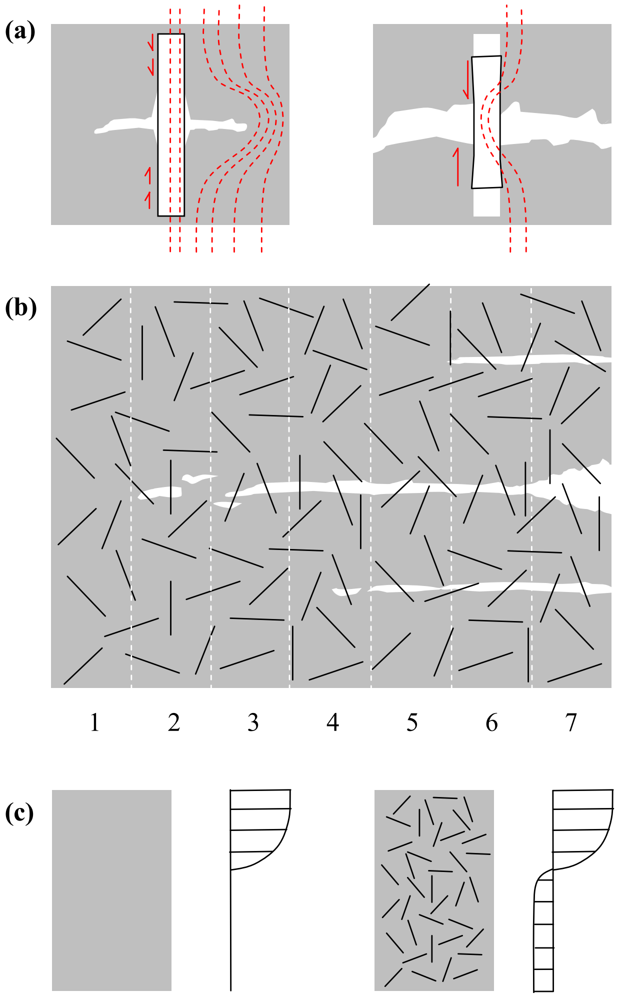

Fig. 1. Schematic overview of the load transfer through a fiber (a), the formation of multiple cracks (b), and the ideal representation of the stresses in the material (c).

When a crack occurs due to tensile stresses, the fibers are pulled out. But in this way, the tensile forces are relocated through the fiber and the fiber will bridge the crack. This is visualized in Fig. 1a. Fibers can either be partially debonded, transfer the load without debonding or can be pulled out [49]. If the tensile capacity of the fiber exceeds the tensile capacity of the matrix and the embedment is strong enough, the material will be able to take up more forces. This will lead to multiple cracking (Fig. 1b). If we would start from an un-cracked material (stage 1), micro cracks would form if the tensile capacity is reached (stage 2, crack suppression). Microfibers along the crack are pulled out (stage 3, crack stabilization) and are able to take up the load. If the tensile capacity of the microfibers exceeds the tensile capacity of the cementitious matrix, a crack will form at another location (stage 4, crack bridging). The same principle is repeated (stages 5–6) until the fibers are no longer able to take up the tensile stresses. At this point, the fibers are pulled out or rupture (stage 7, failure). So, instead of an incapability of transferring the load over a crack (Fig. 1c left), the fibers are controlling the cracking behavior (Fig. 1c right) and transfer forces over the crack.

The use of fibers may increase the overall strength since the stresses are transferred across the formed cracks [53]. So, when

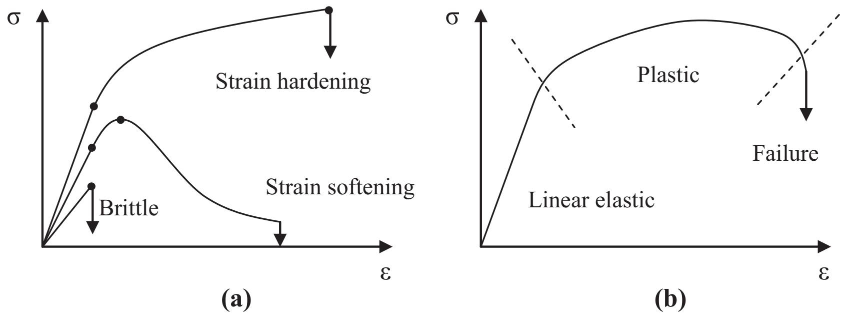

Fig. 2. Three modes of failure with a stress–strain curve (a) and the different subdivisions in the stress–strain curve (b).

the first-cracking-strength is reached, the composite is still able to show additional strain. But this can be divided in strain softening and strain hardening behavior. In the first, the fibers will not be able to transfer additional load and will be pulled out. In the strain hardening case, the stress may alter due to the bridging action of the fibers. The forces which are transferred will be higher. This is shown in Fig. 2. In this figure, three modes of failure are given: the brittle behavior of a plain concrete material, strain softening (quasi-brittle) and strain hardening [54]. A brittle behavior is characterized by a linear stress–strain curve followed by failure (sudden drop). A quasi-brittle material has a softening tail and this behavior is often found in normal fiber reinforced cementitious materials. With strain hardening, there is an elastic linear behavior followed by a plastic behavior until failure. In case of a hardening effect, both sides of a fiber may be pulled out (Fig. 1a) [55], improving the ductile behavior of the composite. Ductility is the deformation capacity under a specific load. Also, the toughness is improved due to debonding and energy absorption. The energy absorption, proportional to the area under the curve, will thus improve for example the earth-quake resistance, impact resistance and the service life of dams [56–59].

By mixing in fibers and designing the material in such a way, the brittle behavior is minimized and the composite will show significant ductile strain-hardening behavior. Several terms are addressing the material in literature. These are: FRC(C) (fiber reinforced concrete/cementitious composites), TRC (textile reinforced concrete), ECC (engineered cementitious composites), HPFRCC (high performance fiber reinforced cementitious composites), SHCC (strain hardening cementitious composites) and MFCFRCC (multiple fine cracking fiber reinforced cementitious composites) [54], among others. The general research in literature has mainly focused on the use of synthetic microfibers, in particularly the polypropylene and polyvinyl alcohol fiber.

2.4. Synthetic microfibers with modifiable properties

In the following part, only the monofilament synthetic fibers are discussed as they are mostly un-influenced by degradation and corrosion and possess high multiple cracking behavior. Several synthetic fibers with their properties are listed in Table 1 [1,4,24,50,60]. In the table, acrylic and polyester fibers may degrade in an alkaline environment. The most used for receiving multiple cracking behavior are the polypropylene fiber and the polyvinylalcohol fiber. These two types of fibers will be addressed in detail.

Polypropylene fibers have a high melting point (165 °C), a low manufacturing price (melt spinning) and a low bond with the cementitious matrix. Monofilament fibers are produced by extrusion, by pushing the polypropylene resin through a circular die. By doing so, the polymer crystals are aligned in one direction. This anisotropy is advantageous because the fibers are now strongly orientated in one direction and are thus stronger in that direction. Afterwards, the long fiber is cut into fibers with the appropriate length. The low bond with the matrix is beneficial as the fibers are thus pulled out more easily instead of being ruptured after cracking the cementitious composite. The low bond is due to the hydrophobic chemical structure of polypropylene. The fibers have, however, a low modulus of elasticity (1–8 GPa). Therefore, high-tenacity fibers were developed with a high modulus of elasticity (up to 11 GPa) and a high strength (*italic* values in Table 1). High tenacity polypropylene fibers consist out of a core (high tensile strength) and an outer layer (improved surface properties) [50,60]. Due to its high elongation, polypropylene is mainly used to restrain the plastic shrinkage at early ages. In that period, the modulus of elasticity of the fresh mixture is that of the fibers. When the mixture is hardening, the modulus of the matrix will become higher than the fibers, making them useless. But at the same time, micro cracks are prevented, reducing the plastic shrinkage and possible future macro cracking. Another use of the polypropylene fiber is for stress release during fire, as they melt during fire and create channels for relieving internal pressure [5].

Polyvinyl alcohol fibers may not be thermo-plastically processed as the polypropylene fiber is and are processed by wet spinning. In this process, the resin is dissolved in water and spun in a salt-rich bath in which the polymer coagulates. Afterwards, there is a procedure of stretching and heat treatment to receive the needed strength. Polyvinyl alcohol fibers have OH^- groups affecting the bond with the matrix. This hydrophilic nature ensures a

Table 1
Several types of synthetic fibers with their diameter [μm], density [$t\ m^{-3}$], tensile strength [MPa], modulus of elasticity [GPa], elongation at rupture [%] and bond [MPa] in comparison to the cementitious matrix.

Type of fiber	Diameter [μm]	Density [$t\ m^{-3}$]	Strength [MPa]	Modulus of elasticity [GPa]	Elongation [%]	Bond [MPa]
Cementitious matrix	–	2.5	3–7	10–40	0.02	–
Acrylic	20–350	1.16–1.18	200–600	5–10	2–50	
Aramid (Kevlar)	10–12	1.44	2000–3000	70–130	2–5	4.5
Nylon	23–400	1.14	800–1000	4–6	16–20	0.16
Polyester	10–200	1.34–1.39	200–1200	10–18	10–50	
Polyethylene	10–1000	0.92–0.96	80–600	5–31	3–100	0.11–0.63
Polypropylene	20–400	0.9–0.95	100–*928*	1–*11*	8–25	0.2–1
Polyvinyl alcohol	10–650	1.3	1200–*1600*	20–*40*	5.7	<2 (coated)

strong bond with the matrix but also a lower dispersion during mixing [61,62]. Due to the strong bond with the matrix, the fibers will rupture instead of being pulled out. This is not beneficial for multiple cracking. A total of 4–6 v/v(%) of fibers needs to be mixed into receive multiple cracking, but this amount is not workable [63]. Therefore, the fibers can be surface treated with oil, to lower the hydrophilic nature [49]. A coating of 1.2% of oil (*italic* values in Table 1) is the optimal treatment for 2 v/v(%) of polyvinyl alcohol fibers. This will enhance the performance since the polyvinyl alcohol fiber both has higher strength and higher modulus of elasticity in comparison to a polypropylene fiber and the modulus of elasticity is approximately the same as for the cementitious matrix (Table 1). Also, the polyvinyl alcohol fiber has a better cracking restriction because the high elongation of the polypropylene fiber is not efficient.

Both these fibers are used as an additive of cementitious materials for optimal use of the so-called autogenous healing.

3. The biomimicry of healing a crack in a cementitious composite

Nature has always inspired humans. Trees are the example for efficient weight transfer to foundations of high rise buildings, a thistle for the Velcro hook-and-loop fastener, a gecko for adhesive properties, a lotus flower for hydrophobicity and non-wettable textiles. This biomimicry can also be found in self-healing cementitious materials. Consider the human power to heal itself of broken bones and cuts. The regenerated bones and tissue are nearly as strong as the original material. If this feature to heal cracks would be designed in cementitious materials, the costs for repair and maintenance could be reduced. Self-healing materials can repair damage without any external intervention. This feature is often called ‘smart’ as the structure needs to monitor and repair the damage. The self-healing mechanism should be pervasive (ready for activation where needed), stabile (active for the service life of a structure), economical (usable for large-scale production, i.e. not too expensive), compatible (no negative effect on the composites properties), reliable (usable in several environmental conditions), qualitative (mechanical properties as good as the plain material) and repeatable (multiple damage events can be healed) [50,64,65].

The self-sealing of cracks was already seen in the beginning of the 20th century as formed cracks in a bridge deck close due to a white precipitation [66]. This is called autogenous healing as cracks close due to material formed by the cementitious matrix itself. Healing can also be autonomic, by incorporating self-healing mechanisms inside the concrete material. In the latter case, the material to close a crack does not originate from the matrix itself, but from another source. Good reviews on self-healing can be found in [67–72] and in the book [73].

Generally, there is a subdivision in self-sealing and self-healing. Self-sealing states the closure of a crack, but possibly without strength regain. Self-healing includes additionally the regain in mechanical properties. Self-sealing approaches prevent the ingress of harsh chemical substances which may deteriorate the concrete matrix. This can be achieved by autonomous healing (e.g. inductive melting of a film pipe, release of encapsulated healing agents, use of shrinkable polymers or calcium carbonate precipitation by bacteria) and autogenous healing of concrete cracks (e.g. further cement hydration, calcium carbonate precipitation originating from components from the cementitious matrix) (as will be explained in the next paragraph). The autonomous healing efficiency depends on the crack width (availability of healing agents), the way of encapsulation (spherical or tubular capsules) and the amount of deposited reaction products (improved by incorporating calcium carbonate precipitating bacteria). The autogenous healing efficiency depends also on the amount of deposited reaction products (improved by matrix tailoring), but also on the availability of water (improved by the inclusion of superabsorbent polymers) and the crack width (restricted by adding microfibers). The self-sealing efficiency is generally evaluated by measuring the decrease in water permeability and air flow through a crack.

Self-healing includes the regain in mechanical properties after crack formation. The extent of regain depends on the type of additive, mix composition and the synergetic interaction between all components. The healing efficiency is usually evaluated by reloading the healed specimen and comparing the obtained mechanical properties with the original ones.

3.1. Autonomous crack healing

Autonomic healing can be by the use of a repair agent in a film pipe which melts under heating [74]. The tube in the latter is made out of a thermoplastic film and the healing agent superglue is encapsulated herein. When a crack occurs, there will be an increased electrical resistance due to the breakage of the conductive path. This will cause a selective heating of the pipe and thus the release of the healing agent which fills the crack. The disadvantage is the embedment of the heating device and the external power supply. Other from outside activated mechanisms like this inductive heating include laser beam activation and ultrasonically induced friction [74].

Another autonomous healing mechanism is the use of encapsulated chemicals both in spherical and tubular capsules [75–80]. Microsphere embedment is one way to promote self-healing. The spheres are uniformly distributed in the matrix and have a thin shell which can be easily broken when a crack occurs in the composite. In this way, the healing agent inside the spheres will come out. The system can be composed out of a one-component or a two-component agent which needs to polymerize after contact with one another. The general principle of spherical two-component encapsulation is shown in Fig. 3 [81]. An encapsulated healing agent and a solid chemical catalyst are dispersed in an epoxy matrix. If a crack runs through the capsules, the healing agent is released due to capillary forces. Exposure of the healing agent to the catalyst triggers polymerization and the crack faces are bound together. Parameters which are important are the thickness of the capsules and the strength (in function of breaking and manufacturing process) and the bonding between the matrix and the capsules [77,78]. Tube embedment is similar to microsphere embedment. The most used material is glass or some other brittle material. A two-component polyurethane as a healing agent may be used for this purpose [82–86] and the crack filling with the foaming healing agent was visualized by means of X-ray computed microtomography (Fig. 4) [87]. Other chemicals may be used as well [88]. Shrinkable polymers, like shape memory alloys which return to their initial shape upon heating, may also be used to close a crack [89]. Glass tubes with one-part epoxy may be used as well [90,91] or a hollow pipe as a core to insert the healing agent [92], mimicking closely the bleeding-based healing mechanism in living things. More closely related to this animal healing is the use of a porous concrete, where the healing product can be inserted after crack formation [93,94]. The bone self-healing process is hereby imitated by putting porous concrete internally in the concrete structure to create a porous network similar to ‘spongious bone’ (Fig. 5).

Micro-organisms that induce calcite precipitation can also promote the self-healing of a crack [95]. This can also be considered as autonomous healing as bacteria, like *Bacillus pasteurii* [96], *Bacillus cohnii* [97] or *Bacillus sphaericus* [98–101], precipitate calcium carbonate to close a crack (as shown in Fig. 6). The general idea is to incorporate dormant bacteria in the concrete matrix. When water

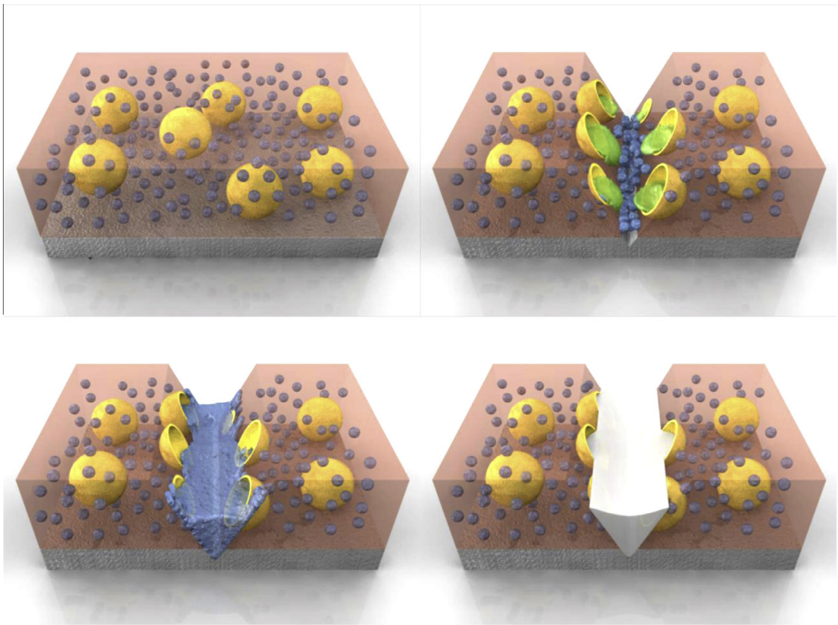

Fig. 3. Autonomous repair of cracks in a composite material: general schematization, after Cho (2006).

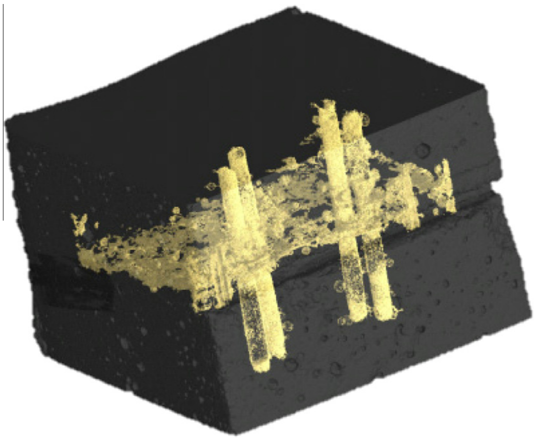

Fig. 4. Two-component polyurethane healing after crack formation visualized by means of X-ray computed microtomography, after Van Tittelboom et al. (2011).

enters a crack, the bacteria will become active, will start to multiply and will start to precipitate calcium carbonate. In case of calcinogenic bacteria, urea is hydrolyzed and degraded to carbonate and ammonium [97]. This is also done by the consumption of organic salts (like lactate) incorporated in the mixture into acetate, calcium carbonate and carbon dioxide. Other bacteria, ureolytic strains, decompose urea into carbon dioxide ions [99,102]. Bacterial calcium carbonate precipitation through denitrification is also possible as the afore-mentioned bacterial mechanisms are sometimes oxygen-limited and toxic side products may form [103]. The carbon dioxide, dissolved in water, has the additional benefit to react with calcium hydroxide to also form calcium carbonate and water. The latter also happens in the case of the autogenous healing method, which will be described in the next paragraph.

A cementitious matrix is not the ideal environment for bacteria as there are no nutrients available. These nutrients also need to be incorporated in the matrix together with the dormant bacteria. The alkaline environment poses no direct treat for alkaliphile bacteria

Fig. 5. Porous concrete healing similar to spongious bone filled with epoxy, after Sangadji and Schlangen (2012).

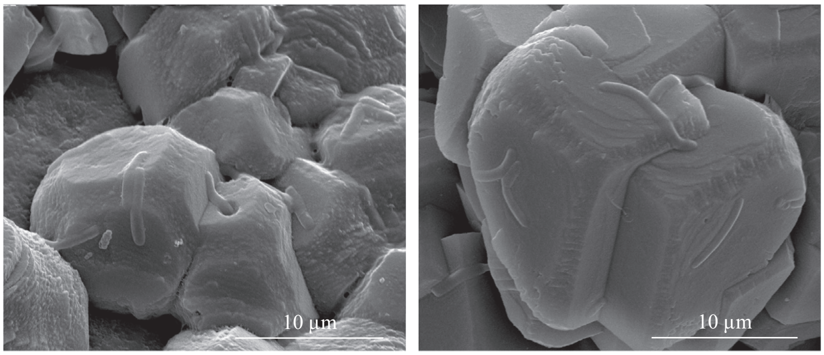

Fig. 6. Scanning electron micrographs showing bacteria and $CaCO_3$ crystallization, after Wang et al. (2012).

which can survive in the concrete matrix. However, the bacteria need to survive the mixing procedure and may not be crushed due to ongoing cement hydration and matrix densification [104]. As a cement pore size of 1 μm is limiting the survivability of the bacteria (with a typical size of several micrometer as seen in Fig. 6) and the pores in a cementitious matrix are of that dimension and below, the bacteria need to be protected. This can be done by immobilization in silica gel or polyurethane [96,98,99], on porous powders (like diatomaceous earth) [105], in microcapsules [106], in hydrogels [107], by incorporation in porous granulates like lava or Argex, or by using air-entraining agents which create larger pores [104]. A strength regain up to 60% is possible [99], but the immobilization reduces the overall strength.

As bacteria precipitate calcium carbonate, bacterial self-healing is often confused with autogenous healing. However, the cementitious matrix is not the only source for this precipitation, thus the mechanism with bacteria should be regarded as autonomous.

3.2. Autogenous crack healing

Concrete has the natural capacity of autogenous crack healing, as first found by the French Academy of Science in 1836 (as stated by [108]). Four main mechanisms and their combined effect contribute to autogenous healing of concrete cracks [50,108–116]:

(a) Dissolved carbon dioxide in water may react with Ca^{2+} ions present in the concrete matrix to form calcium carbonate ($CaCO_3$) crystals.
(b) Loose particles or impurities may block the crack.
(c) Unhydrated cement grains present in the matrix and on the crack surfaces may further hydrate. Also, supplementary cementitious materials such fly ash or slag [117] can further react through pozzolanic or latent-hydraulic activity.
(d) Expansion due to swelling of calcium silicate hydrates (C–S–H).

All four mechanisms are shown in Fig. 7 [109]. The most significant mechanism is the calcium carbonate crystallization [110,111,118]. In this case, a white material fills the crack. With a pH of the water higher than 8, the mechanism of calcium carbonate precipitation is [111,118]:

$$CO_{2(g)} + 2OH^-_{(aq)} \rightarrow CO^{2-}_{3(aq)} + H_2O_{(l)} \tag{1}$$

$$Ca^{2+}_{(aq)} + CO^{2-}_{3(aq)} \rightarrow CaCO_{3(s)} \tag{2}$$

$$CO_2 + 2OH^- + Ca^{2+} \rightarrow CaCO_3 + H_2O \tag{3}$$

For a pH between 7 and 8 [111]:

$$Ca^{2+} + HCO_3^- \rightarrow CaCO_3 + H^+ \tag{4}$$

Its sealing is dependent on the crack width and the water pressure but independent from the type of water [111]. The calcium carbonate crystallization is first surface controlled, but as the Ca^{2+} ions are exhausted in time, the precipitation becomes diffusion controlled. The Ca^{2+} ions then need to migrate through the cementitious matrix [111].

The sealing and healing capacity can be studied by means of a decrease in water permeability in time. In this test, the permeability coefficient is a function of the third power of the crack width [111,119,120]. The sealing of a crack is dependent on the crack width and the hydraulic gradient as the formed healing products may also be washed out [111,119,121–124]. The reduced permeability in cracked specimens is thus due to the combination of mechanical blocking and chemical precipitation of calcium carbonate [125]. As the precipitation of the crystals is promoted by the use of a permeability test [124], one should be critical referring to self-sealing and self-healing in that case.

In high-strength concretes with a low water to cement ratio, the healing is mainly due to the hydration of unhydrated cement grains on the crack surfaces. Using a low water/cement ratio, reservoirs of unhydrated cement grains are dispersed in the matrix, waiting for water to further hydrate. The stiffness of the new crystals is close to that of the primary calcium silicate hydrates [112,126,127]. Also, the younger the material, the more healing will occur due to the higher amount of unhydrated particles. As the cement further hydrates in time, the healing material formed at early ages is a combination of $CaCO_3$, C–S–H and $Ca(OH)_2$. At later ages, the healing material is mainly $CaCO_3$ [109,113]. C–S–H are stronger than the weaker calcium carbonate. This C–S–H is therefore wanted as it will highly contribute to the regain in mechanical properties.

Water is needed in the above-mentioned healing mechanism to heal a specimen [110,112,128]. The crack width and the mix composition seem to be important for the self-healing as they will determine the dispersion of the unhydrated cement grains in the matrix and at the crack faces. Water also needs to be present as the hydraulic (cement) and pozzolanic and latent-hydraulic reactions (binders like fly ash or blast furnace slag) need water. Pozzolans promote further hydration as these materials react with water and $Ca(OH)_2$ to form C–S–H. This mechanism lasts for a long period and is therefore beneficial for possible healing. Puzzolanic fly ash [129,130], blast-furnace slag [131], lime [132] or alkaline activators [133] can be added to receive more autogenous healing. Additives like expandable geo-materials [134] or crystalline admixtures [32,135–138] stimulate the crack healing capacity even further. The expansion will seal a crack and the additives significantly affect the rate of formation of re-hydration products.

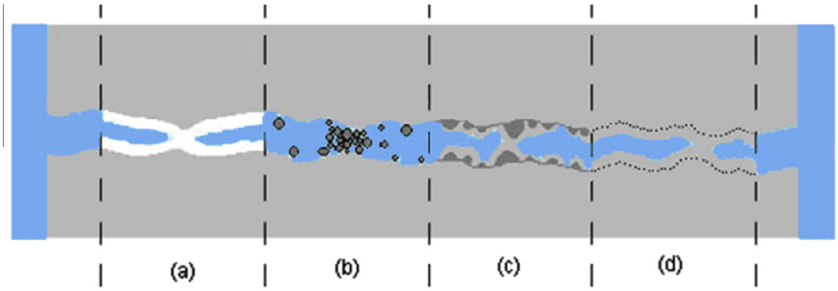

Fig. 7. Different healing mechanisms responsible for autogenous crack healing: crystallization (a), blockage by loose particles (b), further hydration (c) and expansion of calcium silicate hydrates (d), redrawn after ter Heide (2005).

Overall, there are three needed conditions for autogenous crack healing to occur. These are the presence of specific chemical ions (e.g. Ca^{2+}, CO_2, unhydrated particles); the exposure to humid environmental conditions (e.g. wet/dry cycles, submersion in water) and small crack widths (<50 μm) [50,128]. Cracking and permeation inside a crack can induce the needed diffusive forces which can lead the free Ca^{2+} ions to the crack faces. This tendency causes the provision of the chemical buildings stones for the precipitation of healing products. However, the presence of the chemical ions decreases in time and will become exhausted. Water is available during raining and the effect of alternated rainy and dry periods is studied by performing wet/dry cycles. Cracks smaller than 50 μm show complete healing and cracks smaller than 150 μm only show partial healing [50]. From the latter it can be concluded that autogenous healing is only efficient for healing narrow cracks. As the cementitious material has a problem with healing large 'fractures' or 'cuts' like the human body, the crack width should be restricted. The crack width should also be restricted to healable cracks in the frame of corrosion of reinforcements, especially in nuclear applications [139]. This is obtained by mixing in microfibers, as will be described later on.

4. Microfiber-reinforced composites with restricted crack widths

Engineering and nature are dual. In time, nature has developed efficient ways of living using the least amount of resources. This involves an optimal use of energy between the various living organisms. Engineering requests the efficient use of money, to obtain lower costs. If nature is interpreted and mimicked correctly, the efficiency in daily life would improve. The plain cementitious material can be adjusted by the addition of microfibers to receive a cost-efficiently self-healing material. Microfibers are able to control the cracking behavior and to restrict the cracks. Not one large un-healable crack is formed, but several small healable cracks are formed.

4.1. Design of fiber reinforced cementitious composites with polyvinyl alcohol fibers

The main research on fiber reinforced cementitious composites with polypropylene and polyvinyl alcohol fibers was done by V.C. Li. His engineered cementitious composites are able to give tensile strengths of 4.5 MPa, ultimate strains of 4% and limited crack widths of below 100 μm, with only a fiber volume of 2% of the total volume of the cementitious mixture [19,50,63,128,140,141]. The ductility of the composite under four-point-bending is shown in Fig. 8 [54]. Multiple cracking can be observed, which shows the power of this material.

There are two main strain hardening criteria: the first is the crack tip toughness need and the second the force need [50,63,142–144]. The former uses the concept of energy balance and is a statistical analysis of the combination of absorption, debonding, sliding and bridging action. It is a function of the bond with the matrix, the friction and the fiber characteristics. This criterion is for steady state cracking. The net energy input by external work must be less than the energy consumed by bridging during the crack formation. This will be the driving force of crack formation after which fiber bridging will take over [145]. If the criterion is not met, there will be unstable crack formation. This type of

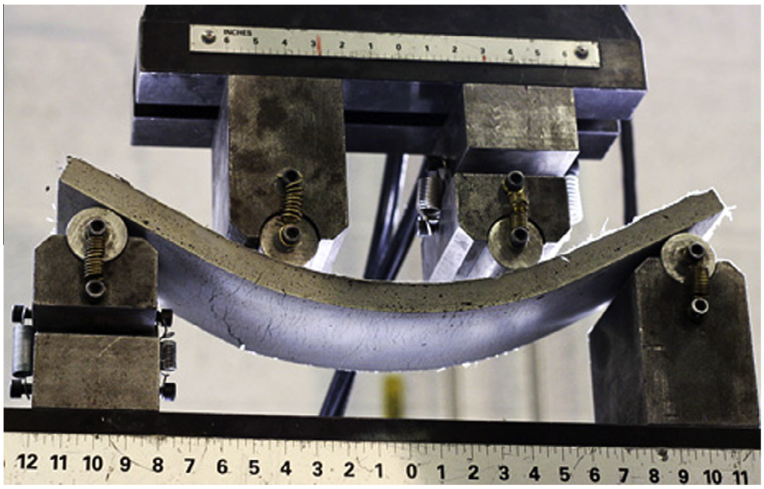

Fig. 8. Multiple cracking and high ductility of the PVA-ECC M45 mixture when loaded under four-point-bending, after Li (2008).

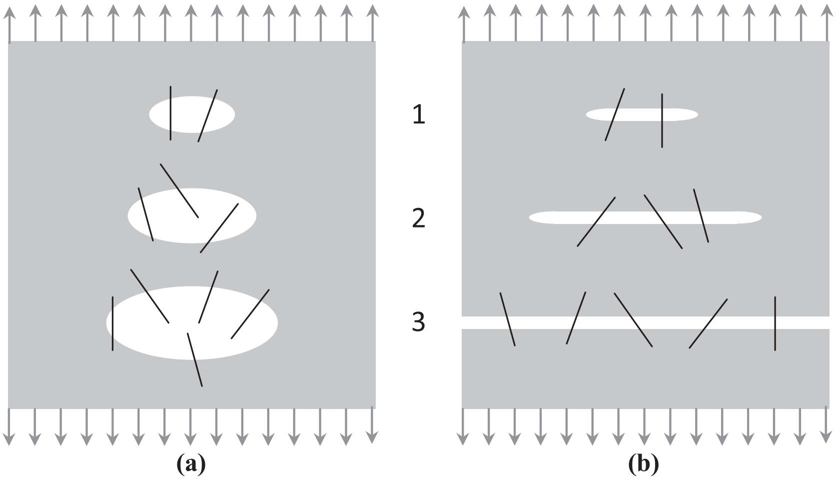

Fig. 9. A Griffith type of crack (a) and a steady-state flat crack (b).

unstable crack is called a Griffith crack and in this type of cracking, the crack width varies over the length of the crack, especially at the midpoint of the crack (Fig. 9) [54]. This will cause fiber failure in that region and is thus unwanted. A stable (steady-state) crack is wanted with a constant crack opening at any location. In this way, there is optimal fiber bridging.

The second criterion states that the matrix tensile cracking strength must be lower than the maximum fiber bridging strength. This criterion ensures the initiation of micro cracks from flaws before the tensile load exceeds the maximum fiber bridging capacity. The time of crack formation is also important as less unhydrated particles are present at later ages [146,147], but the puzzolanic activity increases at the same time. Longer aged cementitious materials lead to more cracks with narrower width [148] as the cementitious matrix is tougher due to further hydration.

Both criteria need to be met and they are a combination of the synergetic interaction between the cementitious matrix, the fiber and the interface. The properties can be altered by the holistic modification of the fiber geometry, treating the fiber surface and optimizing the matrix properties [1]. The matrix toughness can be altered by changing the water to cement ratio, the type of aggregates and the use of supplementary cementitious materials and additives like fly ash, silica fume or blast furnace slag [50,54,149]. The interfacial bond can be altered by changing the fiber surface properties or the type of fiber [50,54]. Also, the length and the diameter of the fiber play a significant role. As long fibers are preferred, they may also cause less workability. The matrix can be tailored by changing the fracture toughness and by introducing flaws. These flaws will cause more cracking and thus higher multiple cracking as they affect the crack tip criterion. Several factors together will thus determine the strain-hardening effect of the composite. In the following part, the most frequently used cementitious strain-hardening composite is investigated.

4.2. Toward a ductile material with a good autogenous healing capacity

PVA-ECC mix 45 (M45) is used often as a typical strain-hardening cementitious composite [50], and its mix composition is shown in Table 2. The fly ash to cement ratio is 1.2, the sand to cement ratio is 0.8 and the water to cement ratio is 0.58 (water to binder ratio of 0.26). Due to the low water to binder ratio, the amount of unhydrated particles is quite high, which can be useful for autogenous healing. The best sand to cement ratio is 0.6–0.8 (sand to binder ratio of about 0.35–0.4) in terms of strain capacity [63]. The sand has an impact on the matrix toughness and interfacial properties. A higher amount increases the toughness due to the increase of energy by the tortuous propagating crack. On the other hand, sand may act as crack initiator but this only has an inferior effect. Sand also increases the interfacial stress and fiber abrasion [63]. This results in less fiber bridging action. The amount thus needs to be limited. The sand particle size is also of utmost importance as the above-mentioned factors are influenced by it [150]. Also, due to the balling effect of fibers together with larger sand particles, the porosity may increase. The ECC M45 mixture uses 12 mm long and 39 μm diameter polyvinyl alcohol fibers coated with 1.2% oil. Due to the hydrophilic nature of polyvinyl alcohol fiber, the bond with the cementitious matrix can be as high as 6 MPa. By coating the fiber with a thin layer of oil [50], the bond is reduced to lower than 2 MPa (Table 1). Coating the fiber with oil affects both of the above-mentioned criteria. In this way, the ductility is enhanced. Some physical properties are listed in Table 3 [54]. The first cracking strength is the strength at which a first drop in the stress strain curve occurs and a complete crack runs through the specimen. The ultimate tensile strength is the highest strength found during strain hardening and the ultimate tensile strain is the strain at which this stress is achieved.

Table 3
Typical tensile properties of strain hardening cementitious composites.

Compressive strength [MPa]	First cracking strength [MPa]	Ultimate tensile strength [MPa]	Ultimate tensile strain [%]	Modulus of elasticity [GPa]	Flexural strength [MPa]
20–95	3–7	4–12	1–8	18–34	10–30

Self-healing of the cracked specimens prevails in a variety of environmental conditions, including conditioning temperature, water permeation and chloride submersion [151]. Water needs to

Table 2
Typical mixture for receiving multiple cracking: PVA-ECC M45.

	Cement [kg m^{-3}]	Fly ash [kg m^{-3}]	Sand [kg m^{-3}]	Water [kg m^{-3}]	Superplasticizer [kg m^{-3}]	PVA-fibers [kg m^{-3}]	Total [kg m^{-3}]
PVA-ECC M45	571	685	456	332	6.8	26	2076.8

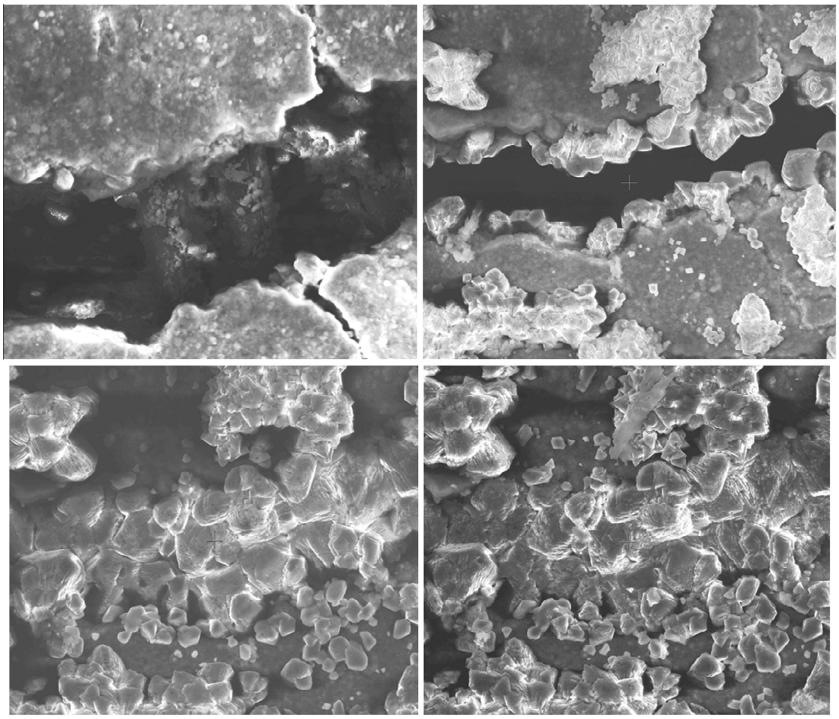

Fig. 10. Autogenous healing of a crack mainly due to calcium carbonate crystallization after several wet/dry cycles, after Jia et al. (2010).

be present, ranging from complete submersion to cyclic wet–dry cycles [128,152]. The best healing and regain in mechanical properties occurred in water/dry cycles (submersion in water at 20 °C for 24 h and drying in laboratory air for 24 h), followed by continuous submersion. A cycle at a certain relative humidity (stored in air) did not provide any form of healing [50,128]. Further hydration of cement grains needs water and the water also encourages the dissolution and leaching of calcium hydroxide from the concrete matrix to form self-healing crystals with the carbon dioxide dissolved in water. The latter has better crystallization possibilities at the crack mouth. An example of this autogenous healing is shown in Fig. 10 [116]. After only several wet/dry cycles, the material is able to self-seal and self-heal. A regain in air tightness is even possible [153].

Autogenous healing can be studied with varied testing methods. The most often used tests are mechanical reloading tests to evaluate the regain in mechanical properties. Renewed tension or bending tests reveal that the majority of the cracks which from tend to follow previous crack lines and propagate trough the self-healing materials. This is due to the relatively weak nature of calcium carbonate crystals [50] and also the weak bond between primary and new-formed calcium silicate hydrate crystals. But, there may also be full recovery of the mechanical properties as cracks may form elsewhere as well [50]. Microscopic analysis supports these findings and is complementary. To compare self-healing in different environmental conditions, the resonance frequency technology is also used recently but is not solid as the matrix around a crack may also change and a weight change in specimens (partly) saturated with water will change the resonant frequency. This will not give objective data to compare self-healing in different environmental conditions. However, this technique is used and one needs to be careful when studying and reporting these results. Therefore, we have to be cautious when comparing the healing effect even using the same evaluation method. Also, in between different countries or laboratories, differences in test conditions apply, such as different healing conditions, different types of water, different imposed water pressure heads, differences in studied cracks (formed by shrinkage, direct tension, flexural tension, and so on) and the different sample age among others.

A self-healing material is often called a smart material. The definition states an interaction with the environment and the ability to respond to changes. Multiple cracks are formed and the material is healed after the imposed deformation. This corresponds with a tree, which will accrete material where the stresses are higher. The material is located in the region where it is needed more. In self-healing materials, the cracks will be closed with additional material too.

Mostly, only the outermost part of a crack is sealed with healing product. The bacterial-based self-healing can be visualized by means of X-ray computed microtomography [154] and this technique can also be used for visualizing autogenous healing. Sealing by precipitation in high-strength low-permeability concrete was already studied by means of micro-focus X-ray CT [155]. It was found that the precipitation occurred only near the surface of the specimen and only the first 0 till 50–200 μm of the 100 μm wide cracks was filled with precipitation. However, in the cited study, the test specimens were stored in the presence of seawater. The obtained healing could thus also be the combination of autogenous healing and salt formation. A recent μCT study [156] gave results on the healing properties of strain-hardening materials. It was found that the extent and rate of healing strongly depended on the initial surface crack width. Also, the region of a crack close to the surface (from 0 to 50–150 μm below the surface) could be sealed quickly with crystalline precipitates. In deeper parts, the healing process takes longer and is more likely continued hydration and puzzolanic reactions. Jonkers [157] showed, in bacterial self-healing concrete, that precipitation mainly occurred near the crack rim leaving major parts of the 150 μm wide crack unhealed. He explained the precipitation at the crack rim due to the relatively high solubility of calcium hydroxide and hypotheses that calcium hydroxide first uses the carbon dioxide from intruding water in the crack, and afterwards the remaining calcium hydroxide would dissolve and diffuse out of the crack into the bulk water. Here it will react with carbon dioxide present near the crack rim resulting in the precipitation of larger quantities of calcium carbonate [135,157].

Bacterial self-healing properties can be included in strain-hardening materials to allow wider cracks to heal [158]. Alkali-resistant bacteria and a food source are hereby included and cracks up to several hundred of micrometers are able to close. Strain-hardening materials, also those containing bacterial healing agents, are also used as patch repair systems [158–161].

A new topic is the investigation of the combined effect of microfibers and superabsorbent polymers (SAPs) [162–165]. As those polymers swell in contact with water after crack formation, the crack is self-sealed [166–170]. The water can then be released toward the cementitious matrix to stimulate the autogenous healing. This water supply complies with the water criterion needed for autogenous healing. The healed specimens are able to regain some of their mechanical properties (up to 75%) [163]. Even second reloading of those healed samples leads to partial additional regain in mechanical properties (up to 66%) [164]. The healing products could also be studied my means of X-ray computed microtomography. The extent of stimulated healing by means of SAPs was substantial [171].

It is even possible to use natural polymers [172,173] or to synthesize the SAP resin in situ [174]. A precursor is hereby injected with initiator and cross-linker. Infrared radiation is used to make the precursor copolymerize. However, this sealing mechanism can only be applied afterwards. By incorporating SAP particles in the mix from the beginning, this can be overcome. SAP particles even act as pre-existing flaws to improve the performance and ductility of strain-hardening cementitious composites [175], further improving the narrow crack width criterion. All effects combined lead to a material which is able to perfectly seal and heal itself (up to 130 μm [163]). Even bacteria can be added to a hydrogel, further improving the overall amount of healing products [107].

5. Future of microfiber-reinforced cementitious composites for autogenous healing

The future of fiber-reinforced cementitious composites for self-healing is key. The possible use of natural fibers may give a low-cost alternative for the synthetic fibers.

Fiber reinforced cementitious composites possess the qualities of a high strength concrete combined with crack width control and energy absorption capacity. Combined with self-healing it is a durable material and very promising to use in the future. However, performance-based durability concepts are still required to get a durability design framework for these strain-hardening materials [176].

The self-healing material needs to be further investigated on its reproducibility (repeatability and reliability) as cracks formed in a second stage will have less chemical ions available at the crack faces for the autogenous repair of the material.

Water is a key parameter next to the restriction in crack width. This needs to be taken into account as not every spot of a concrete structure will be exposed to rain/water. Superabsorbent polymers prove to be useful in this respect since they will promote autogenous healing. In the presence of fluids, the cementitious material is able to heal, further hydrate, increasing its durability. This healing is thus an extension of the life-like quality.

With new composites, new healing agents and new techniques, the self-healing material will only be used more in the future. The role of autogenous healing on corrosion prevention will be important in the future. If cracks are not sealed, chloride-containing water will break down the passive film on the reinforcements. This aspect needs to be considered when autogenous healing is used in real-time structures. The maintenance and longevity of these structures is hereby very important.

One general conclusion can be made; one should continue to build with nature's rules. The bleeding (healing agents), blood cells (microcapsules), blood flow vascular network (porous concrete), blood clothing (polymerization), skeleton and bone healing (crystallization) are only a few properties studied in the field of construction healing. By mimicking nature to enhance performance, more durable constructions will be designed, leading to a higher service life and better overall life quality.

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