

SELF-HEALING OF ENGINEERED CEMENTATIONS COMPOSITES (ECC) IN CONCRETE REPAIR SYSTEM

B NAGESWARA RAO 1*, K BALA CHANDRA 2*, M V NARASIAH 3*, Dr. Y RAMESH BABU 4*

1. *Student, Dept of CIVIL, VELAGA NAGESWARA RAO COLLEGE OF ENGINEERING.*
2. *Dept of CIVIL, VELAGA NAGESWARA RAO COLLEGE OF ENGINEERING.*
3. *HEAD-Dept of CIVIL - VELAGA NAGESWARA RAO COLLEGE OF ENGINEERING.*
4. *Principal - VELAGA NAGESWARA RAO COLLEGE OF ENGINEERING.*

ABSTRACT

Since the concept of high performance concrete was raised in the late 1980's, it is well known that concrete properties have been greatly improved. However, a larger number of existing concrete structures are suffering deterioration resulting from external or internal causes even during the early stage of service life. Durable repair of concrete has thus drawn more attention. To this end, the main objective of this research is to develop and test a novel method for promoting the self-healing behavior in concrete repair system.

A new type high performance fiber reinforced cementations composites called Engineered Cementations Composites (ECC) has been developed in recent years, characterized by high ductility and improved durability due to the multiple micro-cracking behavior. In this study, it was proposed that the original ECC with local waste materials was embedded with capsules to investigate the self-healing potential of this modified ECC material. To realize this self-healing concept, Super Absorbent Polymers (SAP) can be used as the water reservoir enclosed in the capsules, and then provide available water for self-healing process when the capsules are ruptured by cracking. Based on this idea, the preliminary experiments, concerning sealing materials and encapsulation procedure, were first carried out. Three ECC mixtures focusing on the influence of capsule content and capsule size were involved. In order to induce artificial cracks, three-point bending tests have been used to preloaded ECC specimens to different deflection levels. After healing for 28 days, the specimens have been tested again in the three-point set-up.

The experimental results reveal that the recovered deflection capacity of damaged modified ECC specimens can arrive at 65%-95% of control specimens, which is higher than that of specimens without capsule. While the recovery of flexural strength and stiffness rarely show improvement. Compared to the case of coarse capsules, the specimens with fine capsules show more reasonable performance on mechanical properties including the deflection capacity, the flexural strength and stiffness as well as their recoveries. The nano-CT investigation confirmed that moisture transportation took place in more than half of capsules. Under EDX observation, the relatively high concentration of calcium on the crack surface could be considered that the healing product was probably presented in terms of hydration product such as calcium hydroxide or calcium silicate hydrate. However no apparent healed crack was found under ESEM investigation, thus it could be inferred that the healing efficiency was not remarkable in this study. For further research, it is suggested to investigate the contribution of the optimal capsule size and the sufficient water supplement to more effective self-healing behavior.

INTRODUCTION

Problem definition

Concrete is a strong, versatile and economical material that has been widely utilized for constructions over the world. Since the concept of high performance concrete was raised in the late 1980's, concrete properties have been greatly improved. However, a large number of existing concrete structures, such as bridge decks and pavements are suffering deterioration resulting from external and internal causes even during the early stage of service life. In the civil engineering sector of the Netherlands, premature failure of structures leads to a situation where about half the budget is spent annually on maintenance and repair. For concrete structures in particular, 90% of the repair works focus on repair of cracks caused by reinforcement corrosion [1]. Due to the brittleness of concrete, cracking is unavoidable in concrete structures. Cracking can introduce chlorides, sulphates, oxygen, alkali or moisture into the concrete and accelerate further deterioration of the whole system. To address this problem, the durability of concrete repairs has drawn more attention. Even though the quality of concrete repairs has increased a lot in recent years, realizing durable repairs is still difficult.

As stated above, people have tried to make better and stronger materials, which are capable of repairing cracks and restoring their functionality. These materials can be defined as self-healing materials. The starting point for this study is a newly developed class of high performance fiber reinforced cementitious composites with called Engineered Cementitious Composites (ECC), characterized by high ductility, improved durability due to multiple micro-cracking behavior and lower fiber content [2]. Fine fibers guarantee a tight crack width and significant increase in strain capacity. This

ability of ECC to achieve tight crack width can contribute to engage self-healing in a variety of environmental conditions. Therefore, ECC as a means of healing invisible microcracks can prolong the service life of structures meanwhile reduce the maintenance cost. Since ECC has several special properties, the use of ECC for concrete repairs was proposed in the last decade. In a currently running project at Microlab in TU Delft, a number of experiments based on the basic ECC with local available materials have been studied. The preliminary results indicated that a wide range of raw materials could be used as the basis for the ECC-like repair materials [3].

It is well known that fine cracks under favorable moisture conditions has a potential to heal itself, since self-healing phenomenon was first directly observed in cracked water pipes in 1937 [4]. A common agreement is that continued hydration of cement particles within the cracks is one of the main reasons for the self-healing. In fact, a large amount of unhydrated cement is available in most concrete and especially in those concrete with low water/cement ratio. If cracks occur in the matrix and water flow through cracked concrete, then unhydrated cement reacts with it, resulting in new hydration products. This formation and growth of new hydration products will fill in the cracks. Thus, additional water supply at the locations where cracks are formed is highly significant for successful completion of self-healing. The self-healing can enhance the long-term performance of concrete repairs. For these reasons, this project studies the self-healing potential and mechanical properties of ECC material by a novel method.

Objective of the research

Since the ECC material has self-healing potential, the main objective of this research is to develop and test a novel method for promoting self-healing behavior in ECC

materials. The encapsulation approach is considered as novel method of this study. More specifically, it is investigated that when cracks rupture embedded capsules inside the ECC mixture, whether this action can release healing agent (water) for further hydration of cement, without relying on external supply of water. This study will be conducted as a preliminary study to get more insight into a cement-based self-healing coating of old concrete in concrete repair system.

Outline of the thesis

This thesis consists of six chapters. Chapter 1 introduces the motivation, objective and overview of this research. Chapter 2 gives a review of the literature study about the development of cementitious materials and self-healing behavior. Chapter 3 explains the methodology, including the starting point, challenge and approach of the experimental research. Chapter 4 illustrates the experimental set-ups, procedures of the research program. Chapter 5 presents the results from the mechanical tests and microscopic observations followed by the discussions concerning influencing factors and healing efficiency of the self-healing behavior. Chapter 6 summarizes the general conclusions and recommendations for further investigation.

LITERATURE STUDY

This chapter intends to review the previous works on the ECC material including its characteristics and additives in ECC. Moreover, the literature study of self-healing phenomenon in concrete materials including the mechanisms, the conditions and the approaches of self-healing is introduced.

Review of the ECC material

Characteristics of ECC

In the last decades, concrete with increasingly high compressive strength have been applied to civil engineering since modern building constructions rapidly grow towards high-rise and diversity. The addition of steel fiber and powders improves a number of concrete properties. However, most of these materials still remain brittle. In some cases, the brittleness increases as the compressive strength goes up, which poses potential dangers or fracture failures of the concrete. A specially designed cementitious material termed as Engineered Cementitious Composites (ECC) has been developed by Li and continuously evolved over the last twenty years. ECC is characterized by a high ductility in range of 3-7%, a tight crack width of around $60\mu\text{m}$ and relatively low fiber content of 2% or less by volume [3]. In terms of main material constituents, ECC has characteristics similar to regular Fiber Reinforced Concrete (FRC), including water, cement, sand, fiber and some additives. Coarse aggregates are not used because they tend to have negative effects on ductile behavior of the composite. So far, various fiber types and different cementitious matrixes have been used in ECC, but the detail composition of ECC must obey certain principles imposed by micromechanics considerations. The most fundamental mechanical property of ECC is of the ability to carry higher levels of loading after first cracking while undergoing large deformation. The fibers used in ECC are tailored to work with the matrix for the purpose of constraining localized brittle fracture and guaranteeing more uniform distribution of microcracks. Due to the slip-hardening behavior of fibers, ECC can take increasing load that generates new cracks at other sites. It can be observed from Figure 2.1 that first cracking in ECC is followed by increasing stress accompanied by a rise in strain. This strain-

hardening behavior of ECC is similar to ductile metals.

The crack width is another important indicator, reflecting the durability of a concrete structure. ECC exhibits a well crack width self-controlled in terms of a flat steady state microcracks propagation, see Figure 2.2. After the tensile deformation up to around 1% strain, the early microcracks stop widening and remain more or less constant with crack width of around 60µ. ECC material can be tailored to form numerous closely spaced microcracks. The crack width in ECC is much smaller than the typical crack width observed in the reinforced concrete. Moreover, the self-control of crack width can be seen as intrinsic properties of ECC material, rather than depending on steel reinforcement ratio and structural dimensions [7]. Figure 2.2 also shows the tensile strain capacity of 5% that is about 300-500 times great than normal concrete [8].



Figure 2.4 Dry, collapsed and a swollen suspension polymerized SAP particle [10]

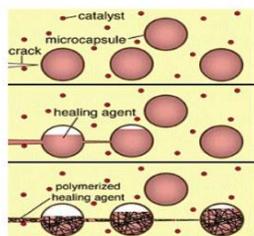
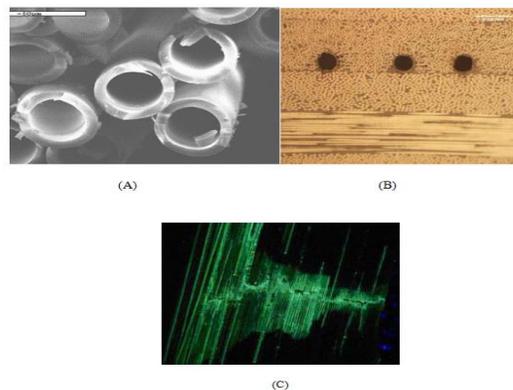


Figure 2.6 Basic method of the microcapsule approach: i) cracks form in the matrix; ii) the crack ruptures the microcapsules, releasing the healing agent into the crack plane through capillary action; iii) the healing agent contacts the catalyst, triggering polymerization that bonds the crack faces closed [12].



METHODOLOGIES

Starting point

The idea of this research comes from an “embedded capsules” approach to repair material itself. Two starting points were proposed in this study to realize the self-healing process in concrete repair system. As mentioned in the introduction, the first starting point is to use ECC material in studying the healing potential. Because ECC exhibits the high strain capacity and tight crack width control, those unique properties can promote the occurrence of self-healing. The second starting point is related to the saturated SAP, here SAP is considered as a water carrier enclosed in capsules since it is able to absorb a large amount of water, and the water released from SAP has the function of promoting the further hydration of the cement.

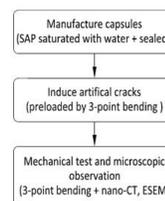


Figure 3.1 Basic approach of the self-healing concept

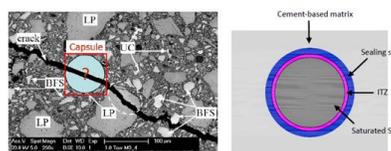


Figure 3.2 Ideal pattern of the crack passing through SAP

Approach

The core question of this thesis can be simply stated as whether water can be released from the embedded capsules thereby promoting self-healing in ECC material. The first task is to find a proper way to seal the saturated SAP. To realize healed cracks in laboratory conditions, the capsules are ruptured by inducing artificial cracks for releasing water. Finally, mechanical test and microscopic observation will be carried out to assess the quality of self-healing. The overview of this approach can be illustrated in the following Figure 3.1.

Figure 3.2 Ideal pattern of the crack passing through SAP

Vi. Self-healing observation

Since the mechanical test until final failure will damage the specimen, how to monitor the internal crack pattern in the case of ensuring the integrity of specimen, that is another problem for this study to assess the quality of self-healing in ECC material.

EXPERIMENTAL STUDY

4.1 Sealing material and manufacture

4.1.1 Introduction

Since further hydration can only be realized in the presence of water or solution, SAP particles can be introduced as a water reservoir in cementations composites. In order to cause the capsules to release the entrained water at the right time, the outer surface of saturated SAP needs to be sealed by a protected layer. In this research, the ideal sealing material can be defined as that which meets the following three requirements. The first is to appear impervious

to leakage of water before inducing the microcracks. Second, this material should be sensitive to cracking, allowing the broken of capsules occurs at a certain level before arriving at ultimate strength of ECC material. Last requirement is the proper interfacial bond strength. This strength of interface between the capsule and the matrix requires being stronger than the strength of the capsule, to guarantee that cracks can propagate through the capsules rather than around them. Thus high bond strength at the interface is one of the important factors contributed to cracks passing through the capsules.

Besides the intrinsic properties of sealing material, the diameter of capsule and the surface morphology also highly influence capsule cracking behavior. Normally the use of capsules embedded in composite materials has a negative effect on the mechanical properties such as strength and ductility, especially when the diameter is relatively larger compared to the specimen size, this disadvantage should therefore be minimized in this research.



Figure 4.2 Manufacture of the capsules

4.1.2 Investigation of sealing materials

4.1.2.1 Selection of sealing materials

In this experiment, two sealing materials were prepared: paraffin wax and epoxy-cement material. The reason for taking advantage of wax is that the wax generally has an excellent water resistant property, stable chemical characteristics. But it is a brittle material that cannot be mixed via a mixer. Moreover the bond capacity of wax could be relatively weaker due to the smooth surface. In this research, keep water available for self-healing behavior is the crucial requirement. Another alternative is to use the epoxy-cement material, since the water-soluble epoxy blended with a number of cement can maintain better compatibility with the surrounding matrix. The composition of this epoxy-cement is presented in Appendix A.

4.1.2.2 Encapsulation procedure

As above mentioned, the size of capsule is not allowed to be large. Here the capsules used in this experiment were made into two groups with an average diameter of 8mm and 5mm, called “coarse capsule” and “fine capsule” respectively. The complete procedure of sealing saturated SAP particles is shown in Figure 4.1. In the first step, every single saturated SAP particle with a small size could be more easily gathered and shaped into a ball when CEM I 52.5N was utilized to form a surface cover. To finish this process, the saturated SAP particles were sieved by 2.4mm size of sieve, to separate them from the excess cement. It is important to control the rate of shaking. If the amount of cement is less, the thin surface cover would not form. However, excess cement will absorb more water from saturated SAP particles. To maximize the contained water inside capsules, one method of avoiding water loss was to cure these balls in water at a temperature of 20 for 7 days, in order to achieve the hydration of cement and keep the SAP particles fully absorbing water. Afterwards, the out surface of ball was sealed by a shell of wax or epoxy-cement,

respectively. For the first case, paraffin wax was heated up to 105°C and then kept a ball into this hot solution for 2 seconds. Finally, wax microsphere was obtained from rapid cooling of the suspension of molten wax droplets and it was cured under room condition. For the second case, 5 wt% epoxy and 100 wt% cement was mixed by hand and then rolled a ball in the epoxy-cement paste until smooth. After this, the ball was cured in RH 100% at 20°C for 7 days. In such a way, saturated SAP particles were made into two types of capsules (Figure 4.2).

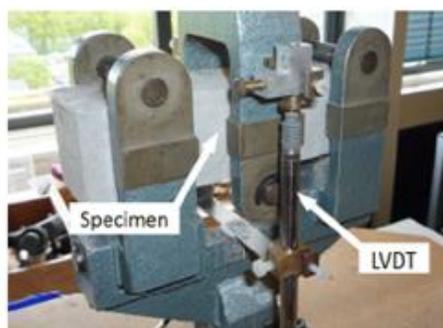


Figure 4.5 Three-point bending test set-up

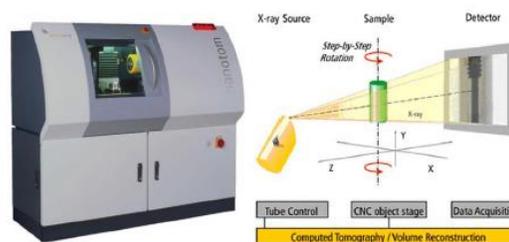


Figure 4.7 Schematic representation of nano-CT system

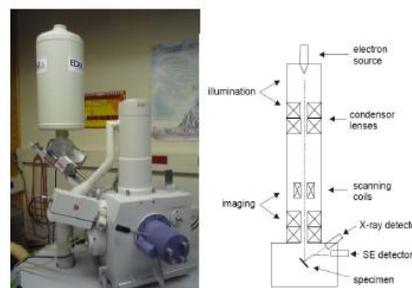


Figure 4.9 Schematic of a scanning electron microscopy



Figure 4.10 Light microscope

Short summary

At the beginning of this chapter, the preliminary experiment for investigating two sealing materials was introduced. Based on using the paraffin wax as the sealing material, the mechanical testing program and several microscopic observations were purposed and introduced to explore the functional performance of healed ECC. The next chapter will present the experimental results and discussions.

Results and Discussion

5.1 Recovered mechanical properties in ECC

Several techniques have been used in examining self-healing behavior. In this section, the self-healing in ECC is evaluated from the point of view of mechanical properties.

5.1.1 Load-displacement relation

The displacement controlled three-point bending test records the load-displacement relationship. One example of load-displacement curve at three different stages of loading is given in Figure 5.1 (A). As indicated, there is an initial linear-elastic part up to the first crack strength. The following is of the propagation of cracks, more microcracks are formed and developed in the specimen but the loading continues to increase during this stage which is called hardening and the material is still capable of resisting higher levels of loading up to a maximum. After the peak load is reached, the applied load becomes to go down, a single

macrocrack has appeared and the material has started to soften.

Compared with the control samples bended until final failure (scheme A), the preloaded samples (schemes B and C) have different stages, as described in Figure 5.1 (B). When the desired preloading level is arrived the specimen then is unloaded. After healing period, the specimen is reloaded under three-point bending test again. Because of reopening of the cracks resulted from the preloading stage, the load-displacement curve of reloading presents differences in terms of deflection, stress, and stiffness, which are discussed further below respectively. The calculation of conversion from load-displacement relation stress-deflection curve is explained in Appendix D.

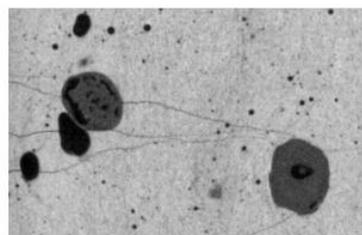


Figure 5.10 Explanation of 2D nano-CT images

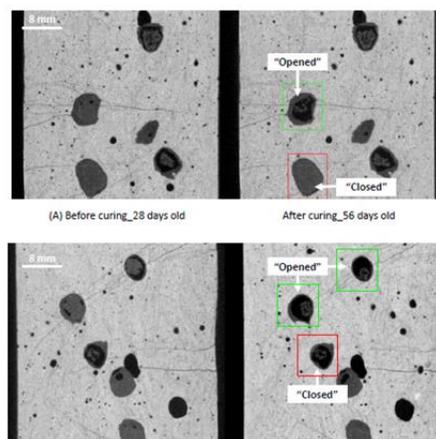


Figure 5.11 Two examples for comparison of nano-CT images before and after 28 days' curing

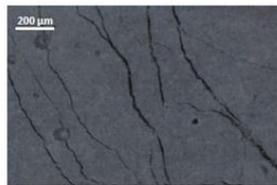


Figure 5.14 Typical crack pattern in ECC specimen

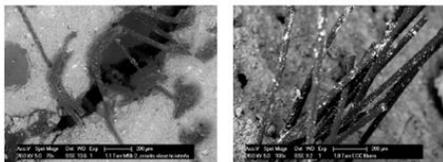


Figure 5.15 Fibers bridging effect in ECC specimen

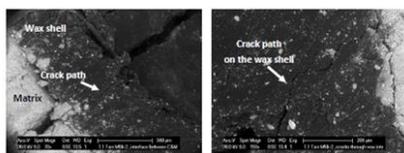


Figure 5.16 Crack pattern on the wax shell

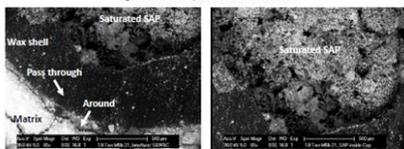


Figure 5.17 Interface between the capsule and the matrix

GENERAL CONCLUSIONS AND RECOMMENDATION

6.1 General conclusions

In this thesis, the self-healing potential of the ECC material by means of available water released from the capsules containing the water saturated SAP has been investigated. Based on the experimental results of mechanical test and microscopic observation, the following conclusions can be drawn,

The mass of water is almost not influenced by the curing time when the paraffin wax is used as sealing material. Due to its better capacity of water storage compared to that of epoxy-cement paste, the paraffin wax was preferred as the sealing material in this research.

Recovery of mechanical properties are regarded as the indicators of the self-healing efficiency. The recovery of deflection capacity, flexural strength and stiffness were examined in this research. The recovered deflection capacity was enhanced while the improvement on flexural strength and stiffness were rarely shown. It could be considered that the self-healing efficiency was not remarkable, since the mechanical properties were not significantly improved.

The ECC specimens with the small capsule size of 5mm in diameter have preferable performance in mechanical properties and their recovery as well as in crack pattern. Similar to normal concrete, the mechanical properties of a cementitious material are highly influenced by the capsule size. Based on the results, the smaller capsule size is conducive to the self-healing behavior.

Under nano-CT technique and ESEM observation, it could be confirmed that moisture transportation took place in more than half of capsules. Sufficient water supplement plays a major role on promoting the self-healing process. In this research, the low efficiency of healing was thus mainly attributed to the insufficient water supply.

The observed healing product probably is of the hydration product such as calcium hydroxide or calcium silicate hydrate. This finding indicates that the self-healing phenomenon probably occurred. Unfortunately no apparent healed crack was observed, it could be concluded that the cracks were likely to undergo the self-healing process, but it is not a very effective healing in this research.

6.2 Recommendations

Several feasible improvements and further research to realize the self-healing of

cementitious materials in concrete repair system can be given as following,

i. Alternative sealing material

As discussed before, a suitable sealing material is not only capable of storing water, but also has high bond strength at the interface between the sealing material and the cementitious matrix. The capsule is considered as the weakest element in the composite and the interface bond of sealing material directly determines on the crack pattern, which influences the release of healing agent. Thus selection of a proper sealing material is crucial as a basis for further study.

ii. Alternative water reservoir

Liapor particle is a promising candidate for carrying water instead of SAP. It has a high water absorption capacity (30%-40% by weight) and the particle is roughly spherical with a diameter of 1-10mm. Therefore, it can be made into a small capsule. As known, the capsule size significantly influences the mechanical properties of the composite. Moreover, control of capsule size is essential to the uniform distribution of capsules and the probability of capsule opening. The application of liapor needs to be studied further.

iii. Encapsulation procedure

From the point of view of producing the capsules, It is important to find an effective method to manufacture capsules quickly and easily. A rolling machine (Figure 6.1) is suggested in future study. This rotary barrel tumbler results in economic, quiet and efficient operation for tumbling small parts [25].

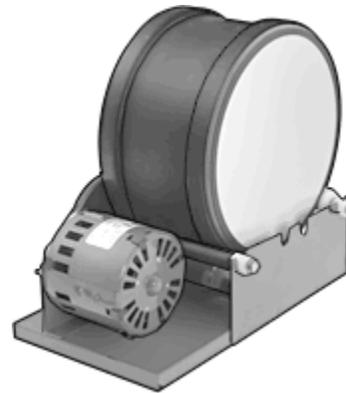


Figure 6.1 Rotary barrel tumbler

iv. Effective healing agent

Currently, sodium silicate used as the microencapsulated healing agent was embedded in a concrete mixture [26]. The sodium silicate reacts with the calcium hydroxide and forms a gel-like material (calcium-silica-hydrate) that will heal the crack and block the pores. The advantage is that the gel hardens in about one week and the recovery of the strength can reach 26 percent of its original strength. It is believed that a more effective healing agent can contribute to the self-healing process.

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