A method for testing self-healing mortar beams with embedded capillary tubes of glue is reported. The findings from a series of trial tests with different amounts of conventional reinforcement, glues, arrangements of tubes and delivery systems are discussed. A revised testing procedure is then described and the results from a first series of tests with this revised arrangement are presented. These show that significant repair occurred in the beams and that the strength, stiffnesses and ductility of the beams were all significantly improved relative to those of the control beams which contained ink filled tubes.

Keywords: Autonomic-healing; self-healing; cementitious; concrete; cyanoacrylate

1 Introduction

The durability of concrete structures has been an issue at the forefront of the minds of engineers and material technologists for many years, due to the fact that the repair and maintenance of structures account for almost 50% of the United Kingdom’s activity in the construction and building industries [1]. Furthermore, in light of the Government’s growing agenda on sustainability, driven largely by worries over global warming, there is a greater demand to produce increasingly maintenance free and energy efficient buildings.

The majority of durability issues in concrete are related to microcracking which occurs due to thermal stresses, creep, drying shrinkage, and general loading under serviceability conditions. These microcracks provide pathways for saline water, acid rain, and carbon dioxide ingress, which lead to chloride attack, reinforcement corrosion, and often spalling damage.

In recent years the issue of material durability has shifted from pure retrospective maintenance regimes towards alternative ‘smart’ solutions, which encompass intelligent materials, smart materials, smart structures and sensory structures, as discussed in [2]. The development of self healing materials is one such technology which has evolved over recent years. These materials may be considered to fall under the category of smart structures since they contain encapsulated healing agents which are released when damage occurs, thereby ‘healing’ the ‘injury’ and increasing the materials’ functional life.

Self healing polymeric materials were studied originally by White et al. [3] and have since been developed extensively by White and his colleagues at the autonomic healing research group, University of Illinois at Urbana Champaign. Unlike polymeric materials, cementitious materials have been observed for many years to have a natural autogenic healing capability, due in part to the re-hydration of previously unhydrated pockets of cement within the matrix on re-submersion within water.
The benefits of autogenic healing are naturally limited, and therefore in recent years autonomic healing has also been studied within the context of concrete by various authors, including Dry [4], Li et al. [5], and Mihashi et al. [6]. Various experimental aspects have been examined by these authors however, to the knowledge of the present authors, no significant numerical modelling work has been completed in relation to the autonomic healing of cementitious materials.

The current work was therefore motivated by the need to develop a numerical model for general self healing processes. As part of this development, and in light of the limited experimental data available, it was considered useful to undertake a series of experiments at Cardiff University, for which all observations and data would be available. As a result, an experimental method has been developed subsequent to overcoming many challenges associated with achieving self healing in laboratory scaled specimens. The solution to the problems encountered and the presentation of sample results of the first successful test series are the main subjects of the present contribution.

It should be noted that full results of the test series are to be presented in a forthcoming journal publication, however, a selection of representative results from one series are presented in this paper.

2 Experimental work

An experimental programme using adhesive filled borosilicate capillary tubes embedded in prismatic cementitious beams was undertaken, similar to that investigated by Li et al. [5] for the case of a fibre reinforced ECC (Engineered Cementitious Composite). Initial investigations involved exploration of various factors including adhesives of different types, viscosities and volumes, capillary tubes of different sizes and configurations, and different preparation procedures for the specimens. In addition, the performance of internal and external supply systems was examined and these two types of delivery systems compared.

It was decided to use a prismatic mortar beam reinforced with a single high yield bar. Trial tests were conducted with 6mm and 3.15mm bars, with relatively small bars being chosen because they produced a distinct crack. It is noted that the reinforcement percentage with 3.15mm bars is close to the minimum EC2 code requirement of 0.15%. A 5mm notch was sawn from the lower edge at the centre of the beam to preset the position of the main flexural crack. All beams were simply supported with a span of 200mm and subject to a central point load and tested under machine stroke control. The trial beam is illustrated in Figure 1.
2.1 Preliminary investigations

The main healing agents which have been proposed to date in the literature are epoxy resins, cyanacrylatates, and alkali-silica solutions. In contrast to the specialist healing agents employed in polymers, these healing agents are generally ‘off the shelf’ agents whose large productivity and therefore relatively low cost are important assets when considering application to a large bulk material such as concrete. A suitable healing agent must also have sufficiently low viscosity to allow migration into the cracks and sufficient strength on setting to resist crack re-opening.

Low viscosity epoxy resins such as Tecroc epoxy injection grout (Tecroc Products Ltd.) currently form the principle healing agent used in the post-damage ‘active’ remediation of critical concrete floors and bridge decks. Epoxy resins, however, are either two part or one part heat activated compounds which make them unsuitable for encapsulation within concrete. Preliminary experimentation has shown that the mixed epoxy compound hardens even in the absence of oxygen, and encapsulation of both compounds separately within the beams results in poor mixing and hence strength gain during healing.

Mihashi et al. [6] present the use of a diluted and undiluted alkali-silica solution as a healing agent in concrete. The alkali-silica solution in the presence of oxygen causes hydration and thus bonding of the original crack faces. The strength of the bond is less than that of glue, but alkali-silica solutions offer good compatibility with cementitious materials, however, the longevity of encapsulated solutions and the healing time following release require further investigation.

Cyanoacrylates (superglues) are one part systems that react to the presence of moisture and are noted for their ability to cure rapidly (in seconds) and provide a bond strength that often exceeds the strength of the substrate, certainly in the case of concrete. They also have very low viscosities, <10 centipose (cPs), and therefore possess the ability to heal cracks less than 100 microns wide. An important property of cyanoacrylates in relation to their use in concrete is the fact that they are acidic solutions. Contact with concrete, which is an alkaline environment, results in neutralisation of the glue and thus potentially quicker setting times. This quicker gain of bond strength can be beneficial in rapid cyclic loading conditions; however, if the setting time is too quick, the dispersion of the healing agent within the crack may be insufficient. Preliminary investigations have shown, however, that the setting time within concrete is sufficiently long to allow the flow of adhesive to occur. This is probably due to the limited moisture content of the 28 day dry cured specimens and the limited exposure to atmospheric moisture. Cyanoacrylate has therefore been used as the sole healing agent within this self healing experimental series.

The most appealing delivery system is that which involves glue encapsulated in microspheres which are embedded in the matrix [3]. However there are many problems yet to be solved before these can be used effectively in cementitious composite materials and therefore embedded capillary tubes were employed here in a manner similar to Li et al [5].

A series of trial tests with were conducted with tubes of 0.8mm, 1.5mm and 3mm internal diameter. These trials resulted in the choice of the 3mm diameter tubes because the smaller tubes proved to be more fragile and difficult to embed in the mix without them breaking. Also, the higher capillary forces on the glue in the smaller tubes meant that a greater percentage of glue remained in the tubes after breakage.

A number of techniques were explored for filling and sealing the tubes, but the most effective procedures were filling using a syringe and sealing with wax.

Two techniques were tried for embedding the capillary tubes during casting. The first was to use a wire frame to hold the tubes in position whilst the mortar was poured and the second was to cast the beam in layers, with the tubes being placed on top of intermediate layers.
The latter technique proved easier to undertake and avoided broken tubes which were common when the former method was applied.

The preliminary series of experiments were conducted using sets of 100mm long tubes embedded in single or double layers, with 5 tubes in each layer. Figure 1 illustrates a beam with a single layer of tubes but in the beams with two layers, the layers were positioned at 20mm and 35mm from the underside of the beam. In each of the two trial series there was one control beam test in which ink rather than glue was placed in the tubes.

There was evidence of a small amount of healing in one test with two layers of tubes but overall it was concluded that the glue had not been drawn into the crack sufficiently to cause significant healing. The limited extent of ink penetration into the cracks is illustrated in figure 2. It is likely that the glue, which had a higher viscosity than the ink, would have penetrated even less. It was observed after the test, when the beam had been broken, that significant quantities of liquid glue and ink remained within the tubes. Figure 2 also illustrates the difficulty in accurately placing tubes manually during casting.

As a result of the above observations it was decided that embedded tubes with open ends would be better and would eliminate the suction effects of the closed ends. Setting of the glue at the open ends of the tubes was not envisaged to be a problem, but trials undertaken to confirm this showed that glue remains unset in the tubes for at least 5 days (the limit of the trial).

![Figure 2: Ink staining of crack faces of control specimens from preliminary experiments](image)

### 2.2 Experimental procedure and results

Following the above preliminary investigations, the following experimental setup was established as offering the most suitable procedure for examining the feasibility of adhesive based self healing within plain mortar prismatic beams.

Four Self-Healing (SH) and two control (C) beams were tested under three-point loading. The testing arrangement, which is illustrated in figure 3, uses a mortar beam specimen with four 300mm long capillary placed in a single layer 20mm from the bottom of the beam.
These tubes were inserted through lubricated pre-drilled holes in the mould end plates. Curved plastic supply tubes were attached to one end of each tube, as shown in figure 4.

The beam was reinforced with a single 3.15mm diameter high yield steel rod, with hooked ends for anchorage, placed on 10mm spacer blocks. The mortar mix was 0.5 : 1 : 3.5 (water : OPC : sand) and was determined to have a mean cube strength at testing of 60N/mm² and tensile cylinder splitting strength of 4.5N/mm².

All specimens were demoulded 24 hours after casting and then cured for 28 days in air. The capillary tubes within the four SH specimens were then filled with cyanoacrylate to a level of 25mm above the centre-line of the straight tube. In order to avoid air voids, glue was injected into the tubes prior to one end being plugged with a wax sealant. Rite Lok EC-5 adhesive produced by Chemence Ltd. was used as the healing agent in all the experiments. This is an ethyl cyanoacrylate based adhesive suitable for bonding a wide range of substrates and possessing a very low viscosity of 5 cPs at room temperature. It is therefore capable of filling cracks down to 0.05mm. It has a full cure time of 24 hours and a tensile strength (ISO 6922) of 20 N/mm². Full details of this adhesive are given in [7]. The two control beams were filled and sealed in a similar fashion, but with an ink tracing die of low viscosity (approximately 3cPs).

A series of three-point bend tests were then completed in a Shimadzu AG-1 test rig fitted with a 20kN load cell, as illustrated in figure 4. All tests were controlled by machine stroke, at a speed of 0.2mm/min. The crack mouth opening displacement (CMOD) and central deflection together with load were recorded on a multi channel Orion data logger.
The self healing beams were loaded beyond the point of first pronounced cracking and then up to a CMOD of approximately 0.55mm. The load was then removed and the beam was left to heal for 24 hours prior to being re-mounted in the test rig and tested to failure or to significant yielding of the reinforcement. The control tests, with ink filled tubes, were tested in a similar manner, except it was unnecessary to allow for any healing period.

The experimental responses of a representative Self-Healed beam and a control beam are given in figure 5.

![Figure 5: (a) Load – Central deflection responses (b) Load – CMOD responses](image)

**3 Discussion and analysis**

Of the four SH beams tested in this series, three showed evidence of significant self healing, as illustrated for a representative case in figure 5. It should be noted that the beam that exhibited limited evidence of self healing was regarded as invalid due to problems with the delivery system.

Both glue and ink were observed to remain in the unbroken halves of the tubes with sealed ends, confirming the findings of the preliminary investigations that sealed tubes prevent sufficient glue being drawn into cracks of these widths.

![Figure 6a: Ink stain. Control specimen](image)
Migration of ink (figure 6a) was observed immediately following the onset of cracking and initial rupture of the capillary tubes, the latter of which was audible during the experiment. Glue was observed to flow out of the crack during the first stage testing and also during the second stage testing, confirming the belief that the cyanoacrylate doesn’t set quickly in the tubes even when exposed to the atmosphere. This gives rise to the possibility of further healing during subsequent testing, although this has not yet been confirmed.

Examination of the cracked faces after testing indicated the presence of glue above and below tube level, as illustrated in figure 6b. This suggests that the glue had migrated upwards into the crack via capillary action of the initial crack as it grew, and also downwards under gravity and capillary action.

One particularly interesting observation is that the responses of the post-healed specimens were stiffer, more ductile and reached higher peak loads than those of the control specimens. This suggests that the glue may have permeated a zone of micro-cracks around the primary crack and caused that zone to be both stronger and more ductile than the original mortar.

The graphs include calculated responses in order to provide points of reference. These calculations were based upon simple beam theory in which it was assumed that the beams were initially elastic and uncracked; became fully cracked when the tensile stress on the lower face reached the tensile strength of the material; were healed in a zone equal in size to the average of those measured from the broken specimens; cracked again when the lower part of this zone reached a tensile stress of 1.25 * the original tensile strength and finally continued until ultimate, as calculated from the EC2 equations with all partial factors removed. The theoretical responses are not intended to provide an accurate model of the tests but rather to provide a check that the experimental responses are between reasonable calculated bounds.

A more accurate model, being prepared at the time of writing, will account for tension softening/stiffening, the increased ductility of the beams, bond slip and strain hardening of the reinforcement.

4 Conclusions

The qualitative and quantitative observations made during the undertaking of an experimental programme on self healing lightly reinforced plain mortar beams subject to three point bending have indicated that an ethyl cyanoacrylate based healing agent supplied via an external supply system is capable of achieving a successful self healing mechanism.
The post-cracked stiffness, peak load and ductility were all observed to increase post healing. Observations made during and after testing clearly indicate that, provided the healing agent is open to the atmosphere and has sufficiently low viscosity, ethyl cyanoacrylate glue is capable of penetrating a significant area of the crack surface under the influence of capillary suction forces and gravity.

5 Future work

Future work will focus initially on the development of a numerical procedure for simulating the healing process. The effect of primary and secondary healing will be examined through three-point bend tests on notched and unnotched specimens containing varying levels of reinforcement. Data from these experiments will be used to calibrate the new numerical model.

REFERENCES