RESILIENT MATERIALS FOR LIFE: BIOMIMETIC SELF-HEALING AND SELF-DIAGNOSING CONCRETES

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ABSTRACT. Material degradation of our civil engineering structures is generally inevitable and consequently structures need regular maintenance. However, collaborative research through the M4L collaboration within the UK has developed a suite of biomimetic self-healing concretes that have the ability to adapt and respond to damage without external intervention; as demonstrated in the first UK full-scale field trials on self-healing concrete. Following on from this pioneering work, we are now facilitating through the RM4L programme the creation of 'smart' materials that can further self-sense, self-diagnose and self-heal when subject to a wider range of damage scenarios. This paper describes work that is being carried out to develop self-healing systems suitable for healing cracks in concrete at multiple scales, and due to time-dependent and cyclic loading, and develop concrete that can self-immunise against both physical damage and chemical attack.

Keywords: Self-healing, Bacteria, Microcapsules. Shape Memory Polymers, Vascular Networks, Self-sensing

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INTRODUCTION

Concrete is used world-wide in infrastructure because of its low cost, high compressive strength, and versatility. The provision of low carbon and sustainable concrete construction is imperative for achieving CO2 reduction targets, which over the life cycle of a structure requires high-performance and durable concrete. Until recently, degradation of concrete was viewed as inevitable, and mitigation has necessitated expensive maintenance. However, a better understanding and knowledge of composites is leading to the creation of a wide range of ‘smart’ concretes, including those with autogenous and autonomic self-healing and self-repairing capabilities. This development is likely to transform infrastructure by embedding self-immunity and resilience so that structures evolve over their lifespan significantly enhancing durability and serviceability, improving safety and reducing maintenance costs.

An earlier paper [1], presented at the 2015 UKIERI Concrete Congress, described current ground-breaking and novel developments in the delivery of multi-scale (~10nm to ~5mm) self-healing concretes as carried out through an EPSRC and industry funded research project, M4L: Materials for Life.

The promising results from this M4L research, as described elsewhere [2], have led to the strategic funding of a much larger research project Resilient Materials for Life (RM4L). Within RM4L, there is a broader remit to focus on tailoring self-healing cementitious systems use in specific commercial applications and on addressing different damage scenarios and conditions. Furthermore, important damage scenarios have been identified including time-related and cyclic damage and chemical damage including corrosion.

In addition to self-healing attributes, RM4L will initiate ground-breaking research aimed at embedding self-sensing, self-diagnosing, self-immunisation and self-reporting capabilities into cementitious systems in order to develop truly biomimetic responses in our infrastructure materials and structures. An overview is shown in Figure 1 below. Both numerical modelling and experimental work will be used in the optimisation and tailoring aspects.

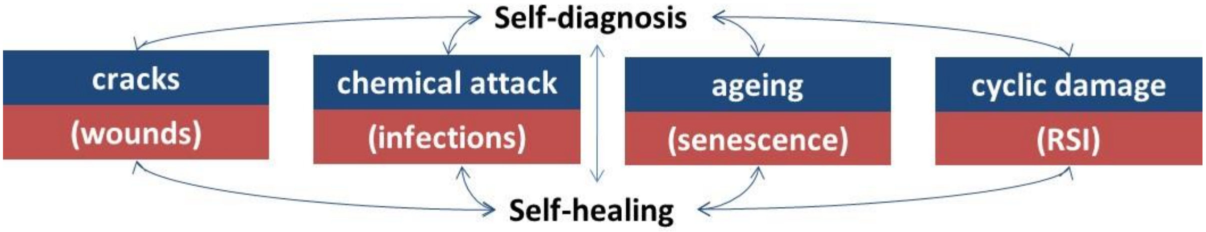

Figure 1 Overview of systems to be investigated in RM4L and their biomimetic equivalent

This paper provides a review of the state-of-the-art in relation to biomimetic self-healing and self-diagnosing concretes of the type being developed and implemented through RM4L.

HEALING AGENTS

In order to achieve autonomic healing in concrete, a wide range of healing agents have been studied over the last 20 years. Healing agents are generally chosen based on their compatibility with the cementitious matrix and time of reaction, although cost and availability of the healing agent are also important factors to consider. In general, healing agents with low viscosity and high sorptivity are usually preferred so as to enable easy flow of the agents into the cracks.

Much basic and fundamental experimental work on self-healing concrete within RM4L and elsewhere utilises cyanoacrylates as the healing agent as they have the capability to cure rapidly in the presence of moisture [3], and have a low viscosity which enables them to heal and seal cracks of widths around 100 μm with few problems. However, cyanoacrylates do tend to form a healing product that is not particularly compatible with concrete and consequently their use in practical applications is potentially limited. The same is true of many other healing polymeric healing agents such as epoxy resins, polyacrylates, polyurethane and methyl methacrylate).

Consequently in work associated with M4L the use of mineral compounds that are more compatible with cement matrices were used; including sodium silicate[4–6], colloidal silica[7-8] and mineral oil [9] and magnesium oxide powder[10-11]. It was observed that colloidal silica and sodium silicate in particular were efficient and consistent as healing agents and can thus be used readily.

The above use of minerals relies on them reacting with the cementitious matrix in the presence of water to create hardened products either via direct hydration or pozzolanic reactions. An alternative that has been much researched is to use bacteria to aid with the precipitation of healing products within the crack. Here bacteria and growth media (carbon and nitrogen sources as well as a calcium salt) are used simultaneously as healing agents. In general, three major pathways for bacteria-based self-healing of concrete have been studied: (i) urea hydrolysis, (ii) aerobic respiration and (iii) nitrate reduction, although there are significant similarities between the three [12].

Research within M4L focussed on aerobic respiration and led to the formulation of a combination of *Bacillus pseudofirmus* (an non-ureolytic) bacteria, nutrients and precursors that will precipitate calcium carbonate in cracks in concrete and return the permeability of the concrete to that prior to cracking [13]. More recent work in RM4L has focused on the selection of new bacterial isolates from the environment for improved healing in concrete [14]. Within this work a high-throughput screening method has been developed to identify isolates that can grow and precipitate calcite in the conditions found in concrete (Figure 2). Initial isolations yielded more than 100 isolates that could grow in alkaline conditions and precipitate calcium carbonate. Further screening yielded 19 isolates for more in depth characterisation, including determination of optimal pH, salt tolerance, and preferred temperature range. Although work in RM4L focusses on non-ureolytic isolates, we have performed a comparison between how ureolytic and non-ureolytic isolates precipitate calcium carbonate in order to understand how these mechanisms differ and how we can apply this knowledge to the improvement of self-healing concrete.

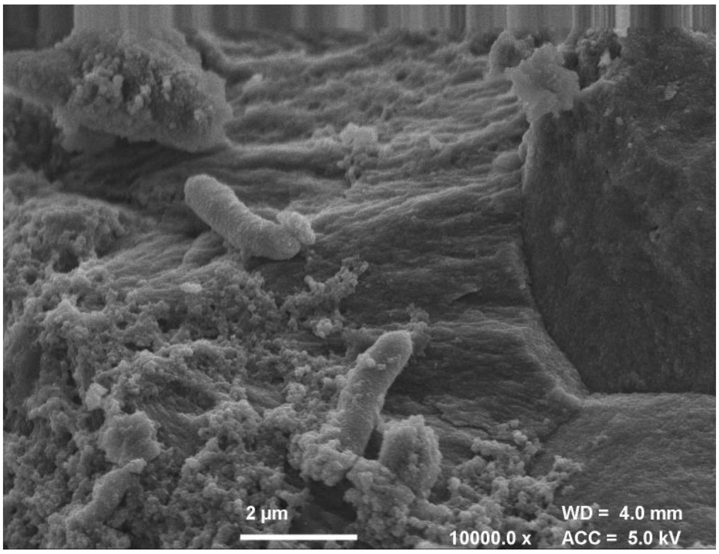

Figure 2 Bacteria induced precipitation of calcium carbonate
(*courtesy of Dr Bianca Reeksting, University of Bath*)

ADDITION OF HEALING AGENTS TO CONCRETE

Autonomic healing relies on having healing agents available to work at any point in the concretes life. It is imperative that they are isolated from the cement matrix so as to not prematurely react. There are two major ways to achieve this: (i) encapsulation and (ii) flow networks. In the encapsulation method the healing agents are enclosed in a protective shell that only permits the healing agent to be released when the conditions are right (usually when cracks form). In the flow network method, the healing agents can be maintained outside of the concrete itself, but flow towards the crack as and when required (Figure 3). A state of the art report produced by the team as part of SARCOS [15] provides a comprehensive overview of the latest work on encapsulation and flow networks. The followings sections relate specifically to M4L/RM4L.

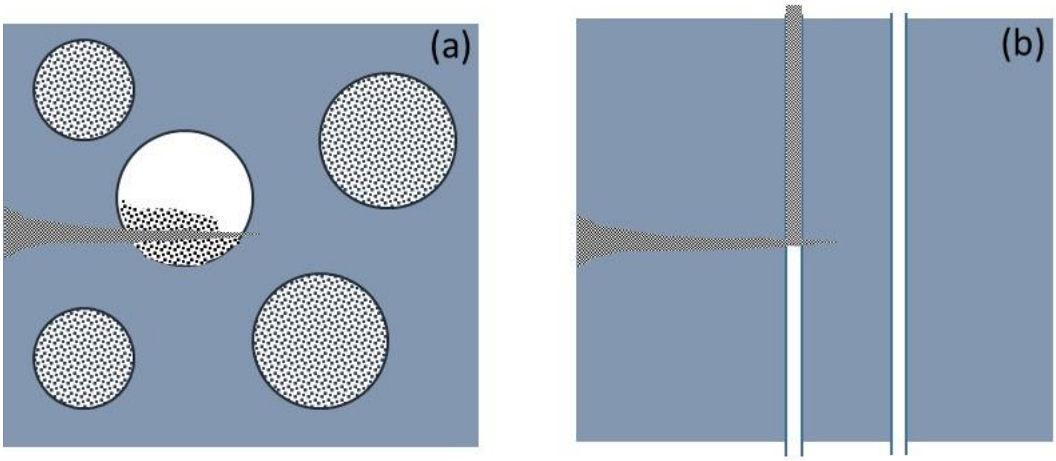

Figure 3 Crack healing in (a) encapsulated and (b) vascular systems

Macroencapsulation in porous materials

Macroencapsulation is a relatively straightforward technological approach to including healing agents in concrete. Here healing agents are added to porous materials, normally lightweight aggregates ranging from 1 to 5 mm in size, under vacuum or by absorption. Research at Cambridge has encapsulated sodium silicate in sintered fly ash (Lytag) [4] and used powder mineral pellets coated in a PVA based film [8]. In both cases, good compatibility with the mortars was shown.

However, within M4L/RM4L, macroencapsulation has most widely been used for delivering bacteria-based self-healing concrete where the bacterial spores and growth media have been enclosed in expanded perlite [16-17]. It has been shown that perlite ruptures on cracking releasing the healing agents in a form that enables calcium carbonate precipitation and healing to take place.

However, a principal problem with macroencapsulation is that the healing agents do not distribute homogeneously throughout the matrix but are concentrated in the specific areas where the capsules lie. Consequently, there are concerns that cracks can entirely miss the capsules should sufficient quantities not be used. The use of larger quantities of macrocapsules to alleviate may not be practical due to the lower strength concrete that would result.

Microencapsulation

Microencapsulated systems in self-healing materials were first researched at the University of Illinois [18] who designed microcapsules that released their contents in response to a stimulus, in the form of mechanical damage, and allowed for multiple simultaneous responses at various locations through dispersion within the matrix. Still world leading, this work now focuses on self-healing in polymers and polymer composites for aerospace applications and microcapsules with a polymeric shell and epoxy core. However, there are further challenges with developing microcapsules for use in cementitious matrices; including stability in a high alkaline environment and survivability during the mechanical mixing without influencing the trigger sensitivity to cracking. Adequate bonding with the matrix is another requirement and healing agents that are more compatible with the cementitious matrix were required.

Research at Cambridge has been investigating microcapsules produced by emulsion polymerisation [5] and microfluidics [9]. Polyurethane shells have also been investigated as these may be better suited for high temperature environments [19]. The use of microfluidics has been particularly promising as it permits surface functionalization and creates a better bond with the matrix. A wide range of healing agents (as described above) have been trialled in microcapsules as described in papers. Furthermore, the size and properties of the microcapsules have been adjusted to obtain optimum healing. Microcapsules ranging in size from 10 μm to 1 mm have been generated and tested.

Collaboration between Cambridge and the company Lambson has led to the design and production of gelatin-acacia gum microcapsules, containing sodium silicate as the healing agent. These microcapsules exhibit initial (during mixing) ductile ‘rubbery’ behaviour and then transition to a stiff, brittle glass-like behaviour [5] in the hardened matrix which enables rupture during crack propagation. Further collaboration between Bath and Lambson has demonstrated the ability to encapsulate spores and growth media in similar microcapsules [20]. Indeed this was the first time that the microencapsulation of growth media for bacteria-based healing had been demonstrated.

Extensive testing on the effect of these microcapsules on the fresh (viscosity, setting time) and hardened properties (E, compressive and flexural strengths) of concrete has been undertaken [4, 6, 21] and the ability of the microcapsules to heal cracks has been verified under different cracking regimes [4, 20]. The microcapsules have been shown to promote healing and recover permeability and sometimes provide a regain in strength – depending on the healing agents encapsulated. Furthermore, it has been shown that microcapsules can be readily up-scaled for full-scale applications [22]. On-going challenges relate to the optimal microcapsule content to achieve an appropriate balance between healing capability and mechanical performance of the concrete. Furthermore, the long-term viability of microcapsules and of the healing agent needs to be proven.

Vascular Systems

Vascular flow network systems are an attempt to simulate the way flora and fauna heal themselves by having an inbuilt flow or vascular network to supply healing agents to the site of damage [23]. A wide range of healing agents can then be pumped through the flow network as required. The reservoir of healing agent can be readily topped up allowing for multiple healing events and the healing agent can be varied to suit the type of damage.

Whilst the use of vascular flow networks for concrete have been looked at by earlier research teams, they tended to form them using brittle glass channels which created difficulties in manufacture as they were likely to break [24]. For this reason, a novel approach has been developed at Cardiff [25]. Here, shape memory or polyurethane tubes are embedded in the concrete and removed after the concrete has hardened around them. This leaves a hollow network within the concrete. Healing agents can then be added remotely to flow and migrate through the network as required (Figure 4). This is best done under pressure once air has been removed from the network. By developing joints within the tubes, Cardiff have demonstrated the ability to produce 2D and 3D vascular networks [25-26]. Further work at Cambridge has developed 2D and 3D biomimetic vascular networks, as shown in Figure 5, employing Murray’s Law and using 3D printed polylactic acid (PLA) [27-28].

Figure 4 Healing of a crack due to flow through an internal vascular network [26]

Figure 5 3D biomimetic vascular networks developed at the University of Cambridge [27-28]

CRACK CLOSURE TO AID SELF-HEALING

The encapsulation and vascular network healing methods described above are more efficient the smaller the crack size. Therefore there has been interest in exploring parallel systems that can limit the crack size and optimise these processes. One approach involves incorporating reinforcement into cementitious structures that can either restrict crack growth or provide crack closure so as to enhance the autogenous healing that will take place naturally. Engineered cementitious composites (ECCs) using high dosages of polypropylene fibres have been shown to limit the growth of cracks and to provide the composite with an inherent self-healing capacity in typical natural environments [29]. However, a novel approach is to incorporate shape memory materials that can reduce crack size after damage has occurred. The shape memory materials are embedded and anchored to the concrete in a temporarily extended form. Upon cracking the tendons are activated and return to their original length providing a compression that confines the concrete. The state-of-the-art on the use of shape memory materials in self-healing cementitious materials has been provided in a comprehensive literature review of which RM4L members were co-authors [30].

Early work by the team at Cardiff investigated shape memory alloys [31], whilst more recent work is considering shape memory polymers (SMP) which are potentially more cost-effective [32-33]. The shape memory properties of SMP relate to the material structure, morphology, manufacturing process and the programming regime. For the SMP used in M4L research the shape memory properties are generated during the drawing process and they have a thermos-responsive property that enables them to return to their permanent shape when subject to heat. As the closure of the crack by the SMP is due to the restrained shrinkage stress, on-going work is aiming to optimise this. Here it is necessary to investigate the effects of draw ratio, draw rate, the degree of crystallinity and the orientation.

SMP multi-strand sheaved tendons have been developed and have been shown to be able to close cracks in the concrete [34]. Further research is considering that the shrinkage stress developed by the SMP could be utilised to counter creep strains in concrete structures. This would reduce long-term deflections and generate longer serviceability lives.

SELF-SENSING OF PHYSICAL DAMAGE

The capability for concrete to self-sense damage and initiate a healing response was not investigated in M4L and is an entirely new research area within RM4L. Indeed, as far as we are aware there has been no research where autonomic self-healing responses have been triggered by sensors embedded within concrete. That said, there has been much research worldwide into self-sensing concrete as a method for early-age concrete monitoring, impact detection and structural health monitoring. There are two key approaches to self-sensing: (i) non-intrinsic sensing which utilises: strain gauges, piezoelectric materials, shape memory alloys or polymer composites, and (ii) intrinsic self-sensing, sometimes referred to as self-monitoring concrete, which utilises functional additives. In a recent overview the main factors that affect the performance of self-sensing concrete have been identified [35].

Piezoelectric materials are popular sensors due to their ability to detect a wide range of parameters (stress, temperature, cracks and damage) and due to their high sensitivity and stability. Joint work between Bath and IIT Delhi has reported some success in the use of piezoelectric materials in cementitious materials for detecting intrinsic changes in the material itself and successfully demonstrated the ability of rebar mounted piezo patches to detect and monitor rebar corrosion and the development of cracks in the adjacent concrete material [36]. Initial work within RM4L has shown that piezoelectric sensors specifically designed for the specifics of concrete have the ability to detect small cracks over much greater differences than "off-the-shelf" sensors [37-38]. Other work at Bath is investigating the use of impedance spectroscopy (dielectric spectroscopy) using both paired electrodes and electrode arrays to evaluate both physical and chemical changes in cementitious materials.

Certainly, an area of increasingly significant research is intrinsic self-sensing. Here functional fillers such as carbon fibres, carbon nanotubes or graphene are added to concrete to fundamentally change the conductive network within the concrete. At Bath we have recently been carrying out research using conductive fillers to alter the resistivity of concrete for the purposes of electrical impedance tomographic imaging and have made some progress on these issues. Work at Cambridge has been initiated on the development of graphene-cement composites for self-sensing with the use of a range of materials including graphene oxide, graphene nanoplatelets and graphite(Figure 6); addressing one of the main challenges in their application, that of effective dispersion in cementitious matrices [39-40].

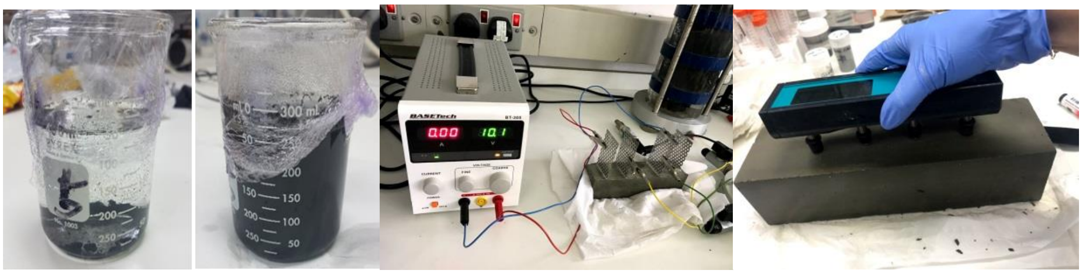

Figure 6 Graphene-cement composites developed at University of Cambridge (*courtesy of Ioanna Papanikolaou, University of Cambridge*)

SELF-DIAGNOSING AND SELF-HEALING CHEMICAL DAMAGE

In addition to healing agents targeting physical damage, recent work has started to focus on agents for targeting chemically induced damage and corrosion. This is an entirely new area of research for RM4L. Cement-based infrastructure materials are commonly exposed to a myriad of damaging chemical environments throughout their service life. This exposure is expected to significantly intensify in the future as a result of more complex infrastructure projects and severe environments in which construction will take place e.g. off-shore and nuclear. These deleterious chemical actions include chloride ingress (marine environments), alkali aggregate reactions, carbonation, sulfate attack (in sulfate-rich soil environment), heavy metal and organic contamination (in contaminated soils) and highly acidic or alkaline environments (in waste leachates). The damage can manifest itself as weakened areas of materials, oxidation of reinforcement, loss of rebar cover, material dissolution, swelling and/or expansion as well as internal microcracking. This damage can also lead to significant macro-cracking although in some cases, e.g. corrosion of prestressed tendons inside concrete, damage may not be detected until too late.

While there are diverse and differing forms of chemical attack, a significant number are associated with steel corrosion in reinforced concrete. This includes chloride and CO_2 ingress and pH and electro-chemical changes. Current systems for steel protection are prohibitively expensive for conventional concrete; and on many occasions are ineffective; consequently repair is currently the main way of dealing with chemical damage. While various recent developments of self-healing anti-corrosion coatings for steel, some including microcapsules, are being commercialised, they are not applicable in cementitious systems and are extremely expensive. The development of self-diagnosis, self-immunisation and self-healing systems for cement-based materials is a new area of research and hence requires radical approaches that rely on the collective expertise of the assembled RM4L team. Research activities already started include the development of pH and chloride sensitive microcapsules (Figure 7).

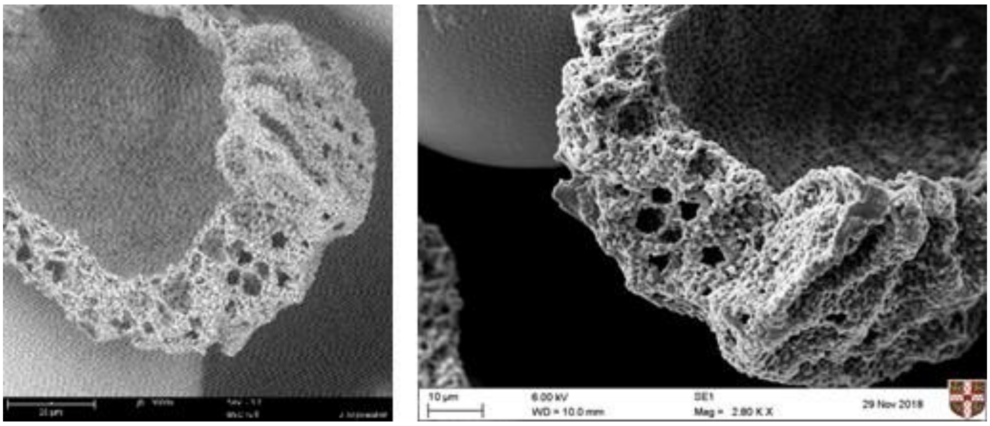

Figure 7 SEM images of pH sensitive micro-capsule shells
(*courtesy of Dr Livia Souza, University of Cambridge*)

NUMERICAL STUDIES

Alongside the above experimental work has been a substantial effort to understand and simulate the processes that underlie self-healing behaviour. In this regard, a number of coupled numerical models have been developed that simulate the transport of healing agents in cracked matrices and the associated mechanical healing of cracks. Such models could potentially be used to guide future material developments. A comprehensive literature review on numerical models for self-healing concrete has been carried out by the RM4L team as part of a SARCOS COST action [30]. It was found that many models were based on limited experimental data and require significant verification, and that the development of a set of reliable comprehensive models is at an early stage.

CLOSING REMARKS

It has been estimated that within the UK the annual cost of repairing our infrastructure (mainly concrete) is £40bn [41] and that the cost of disruption may be an order of magnitude higher again. A principle problem is that damage is very often not noticed until it has become substantial in size and consequently significant deleterious actions can take place before repair occurs. A further problem is the delay that often occurs between diagnosis and repair, as the owner of the structure needs to source or budget for the cost of repair. This can lead to delays of months, or even years, in which significant further damage can happen. The vision of RM4L is to create:

"...a sustainable built environment and infrastructure comprising materials and structures that continually monitor, regulate, adapt and repair themselves without the need for external intervention."

Our research envisages that this can be done by providing our concrete and infrastructure materials with an autonomic capability to respond to cracking, damage and other deleterious actions in a "smart" way. As described above much of our early work through M4L has focused on the provision of a crack self-healing capability. Indeed, our inspiration has been the way in which higher flora and fauna possess an ability to protect themselves against harm and detect and heal damage when it occurs.

The technologies we have developed are capable of healing cracks up to 1 mm in size and providing a recovery of permeability properties and sometimes strength. The healing actions take place immediately upon onset of damage such that recovery of properties is within days or weeks of damage occurring. In general, we have found that there is no single solution to the problem of healing cracks autonomously and that the choice of method to be used, be it (i) encapsulation of minerals, polymers or bacteria, (ii) use of a vascular system or (iii) imposition of a crack control technique, will depend on the specific application, environment and exposure conditions. Through RM4L we aim to demonstrate the use of these technologies in various applications with the intention of providing guidance on the appropriate choice.

Where RM4L differs from M4L, as it was described at the last UKIERI Congress, is with the additional research to model the behaviours that we are observing and provide our concrete and infrastructure materials with the additional abilities to self-sense, self-report and self-diagnose their condition such that they can trigger responses in an "intelligent" way. Consequently our materials will have the ability to become more animate. Potentially these sensing properties could recognise the onset of physical or chemical damage and autonomic responses within the concrete could be stimulated to alleviate these problems before they have actually occurred.

There are enormous opportunities for these self-sensing, self-diagnosing and self-healing concretes as the range of potential applications are wide-ranging. We envisage them being used in coatings and DIY products, precast products, ground remediation, and large scale civil engineering of roads, tunnels, railways and ports. Given the costs related to repair and the associated disruption, the likely annual savings of eliminating manual repair in the UK alone are in the order of £100bn.

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