Aspects of the Behaviour of Engineered Cement Composites

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EXECUTIVE SUMMARY

Engineered Cement Composite (ECC) materials have the potential to be used in civil engineering applications where a level of pseudo-ductility is required. Of particular interest is the possibility of eliminating the steel from reinforced cementitious structures ensuring that no long-term corrosion exists, which is especially relevant for hydraulic tunnels.

Uncertainties remain, however, with regard to the mechanical performance, physical properties, durability and shrinkage of these materials, especially when they are used in thick sections for large scale engineering structures. The current work has studied ECCs in this light. The physical properties such as density, porosity and fibre dispersion and orientation are also of interest: this forms the classic materials engineering triangle of the links between material composition and manufacturing process, the microstructure and mechanical properties.

A cementitious matrix, reinforced with polymeric fibres, has been manufactured using two different processes and fibre types. Specimens have been tested in tension and flexure, and multiple matrix cracking has been observed, which leads to a pseudo-ductile behaviour. Enhanced mechanical performance in tension is in line with a greater fibre alignment and higher levels of porosity do not necessarily lead to a loss of pseudo-ductility. Flexure testing shows a pseudo-ductile behaviour maintained for over three years. The fibre surface coating and the interfacial properties are relatively stable, which is in line with the maintaining of the pseudo-ductility. Theoretical models suggest that the results are in line with the ACK model, particularly for a fibre volume fraction which considers fibre orientation and fibre pull-out is likely to be responsible for the pseudo-ductile behaviour of the material.

ECC materials tend to exhibit a high shrinkage on cure; this could result in cracking, which could compromise the longevity of structures. Methods for controlling shrinkage include the controlling of the environment and use of additives such as powder micro-silica.
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Table 5-3: Interfacial bond properties of specimens aged 7 days (22 tests), 28 days (21 tests), 3 months (20 tests) and 5 months (20 tests)

Table 5-4: Number of each slippage regime mode type per pull-out test age

Table 5-5: Initial Young’s modulus before the opening of existing cracks (E) and after the opening of existing cracks (E*) – N/A meaning that the data is not available
Notation

A: cross-sectional area of the fibre
CS: chemical shrinkage
d₀: distance between the two LVDTs in flexure testing
d₁ and d₂: lateral dimensions of the beam specimen tested in flexure
dₐ: fibre diameter
E: composite Young's modulus before initial damage when being pre-tested in tension
E*: composite Young's modulus after initial damage when being re-tested in tension
Eₐ: composite Young's modulus
Eₐ*: composite Young's modulus taking into consideration fibre orientation
E_{III}: composite Young's modulus in zone III (post-cracking zone)
Eₐ: fibre Young's modulus
Eₘ: matrix Young's modulus
F: applied load in flexure testing
Fₐ: flexural load at first crack
F_{max}: maximum flexural load
g: snubbing factor
Gₐ: chemical bond (debonding) energy
Gₘ: shear modulus of the matrix
h₀: concrete notional thickness depending on specimen size and ambient humidity
Jₐ: crack tip toughness
l: distance between the supporting rollers in flexure testing (the span)
lₑ: fibre embedded length
lₐ: fibre length
\bar{l}_{po}: mean fibre pull-out length
l_{t₀}: initial length of the beam specimen along its 500 mm length
lₜ: length of the beam specimen along its 500 mm length at a time t
Lₐ: stress transfer length
M: bending moment
Mₐ: bending moment at first crack
P: load to pull-out a single fibre
$P_a$: single fibre pull-out load at A (load at which fibre debonding starts)

$P_b$: single fibre pull-out load at B (load at which the fibre is fully debonded)

$PP$: permeable porosity

$R$: mean separation distance between fibres

$R_f$: fibre radius

$S$: shrinkage

$t$: time

$t_0$: concrete age when drying starts

$T_t$: temperature of the specimen at a time $t$

$V$: volume of ECC samples measured using the water displacement technique

$V_C$: volume of hydrated cement

$V_{CI}$: volume of cement before mixing

$V_f$: volume fraction of fibres

$V_f^*$: apparent volume fraction of fibres taking into consideration fibre orientation

$V_{f,cm}$: critical volume fraction of fibres

$V_{hy}$: volume of hydrated products

$V_m$: volume fraction of the matrix

$V_W$: volume of reacted water

$V_{WI}$: volume of water before mixing

$W_b$: buoyant mass of the ECC sample in water

$W_d$: oven dry mass of the ECC sample in air

$W_s$: mass of the water saturated surface-dry ECC sample weighed in air

$x$: transfer length

$x^*$: apparent transfer length taking into consideration fibre orientation

$x_d$: modified transfer length according to Wu and Li (1995)

$x_{d,r}$: modified crack spacing according to Wu and Li (1995)

$x_i$: mean saturation crack spacing

$x_i^*$: apparent mean crack spacing taking into consideration fibre orientation

$\alpha$: coefficient which is a product of several coefficient depending on variables such as fibre distribution, orientation and bond efficient

$\alpha_s$: coefficient of thermal expansion of the ECC sample assimilated to a cement material

$\beta$: shear-lag parameter
\( \beta_s \): factor for shrinkage development with time
\( \delta_0 \): crack opening at the maximum bridging stress
\( \varepsilon \): tensile strain capacity
\( \varepsilon_1 \): shrinkage factor depending on the environment
\( \varepsilon_2 \): shrinkage factor depending on \( h_0 \)
\( \varepsilon_c \): surface compressive strain measured in flexure testing
\( \varepsilon_{c,m} \): corrected surface compressive strain measured in flexure testing
\( \varepsilon_{mu} \): ultimate matrix tensile strain
\( \varepsilon_{muc} \): Composite tensile strain at first crack
\( \varepsilon_{pc} \): maximum post-cracking tensile strain
\( \varepsilon_s(t,t_0) \): magnitude of shrinkage from \( t_0 \) to \( t \)
\( \varepsilon_{s0} \): basic shrinkage coefficient
\( \varepsilon_t \): surface tensile strain measured in flexure testing
\( \varepsilon_{t,m} \): corrected surface tensile strain measured in flexure testing
\( \phi \): curvature (flexure testing)
\( \phi_{max} \): maximum curvature (flexure testing)
\( \Upsilon_m \): matrix fracture energy
\( \lambda \): coefficient which is a product of several coefficient depending on variables such as fibre distribution, orientation and bond efficient
\( \eta_l \): fibre length distribution factor
\( \eta_\theta \): fibre orientation distribution factor
\( \sigma_{b,fc} \): bending stress at first crack
\( \sigma_c \): applied composite stress or composite strength
\( \sigma_{cc} \): first cracking stress of the composite
\( \sigma_{cf} \): flexural strength
\( \sigma_{c,max} \): maximum composite stress
\( \sigma_{cs} \): compressive strength of the material
\( \sigma_{cu} \): ultimate strength of the composite
\( \sigma_f \): stress carried by the fibres or fibre strength
\( \sigma_{fu} \): fibre ultimate strength
\( \sigma_m \): stress carried by the matrix or matrix strength
\( \sigma_{mu} \): matrix ultimate strength
\( \sigma_{pc} \): composite maximum post-cracking stress
\( \sigma_{ss} \): steady-state cracking stress
\( \sigma_{ts} \): tensile strength of the material
\( \tau_0 \): fibre/matrix frictional bond strength
\( \tau_{id} \): interfacial shear strength
Chapter 1 - Introduction

1 Introduction

1.1 Context

Within the Civil Engineering industry, an important branch is that of Underground Construction, i.e. the process of constructing tunnels, shafts and building underground structures within them. Tunnels are built for a variety of purposes, such as transportation (road, rail and water), power services (electricity, gas) and storage (nuclear). Furthermore, tunnels are an environmentally friendly form of construction minimising the impact on the physical environment. Most of these underground structures are intended to be operational over a design life of 150 years and hence ascertaining the durability of the tunnel lining material is an issue of prime importance.

Current tunnel linings are primarily constructed with steel bar reinforced concrete, which can degrade with time. Deterioration arises primarily from corrosion, especially when degradation of concrete cover leaves the steel reinforcement exposed. The use of steel fibre reinforced concrete is now well established in the construction community. However, like traditional reinforcing steel bars, such fibres are prone to corrosion and, due to the small diameter of the fibre, even mild corrosion can represent significant loss of the fibre cross-section leading to an uncertainty of the long-term performance of the material. Therefore, there is within the industry, an interest in alternative material solutions to achieve efficiencies and build more durable structures.

Whilst some current water tunnels operate as gravity pipelines, in the future, such tunnels are more likely to be pressurised due to the increase in population and hence demand for water. Current tunnels are inadequate to cope with a high internal pressure under these circumstances. This issue is exacerbated in water/sewer tunnels, which during operation may well work under tension resulting in the development of full depth cracks. Limiting crack widths is a challenge, especially in hydraulic tunnels, to prevent water and gas ingress and thus compromising the durability of the lining. Hence, alternative material solutions, being also able to withstand a certain level of tensile stress, would be valuable. Furthermore, several of the existing tunnels in the UK have reached a state that require repair to remain serviceable. Examples of one such repair technique would be a permanent lining which includes polymeric
fibres in a reinforced cement composite. In addition to attractive mechanical properties, this type of composite material has significant potential to reduce costs during the whole life cycle of a tunnel by enhancing the durability and by minimising maintenance.

Since 2006, Morgan Sindall Underground Professional Services has been involved in the research and development of a class of materials (for tunnel lining applications) referred to as Engineered Cement Composites (ECCs). This material is a form of High Performance Fibre Reinforced Cement Composite (HPFRCC) and has great potential as a tunnel lining (either as cast-in-place, sprayed or pre-cast).

1.2 Engineered Cement Composite (ECC)

ECCs typically consist of a cementitious matrix reinforced with a relatively low volume fraction of small diameter polymeric fibres (less than 50 µm). Initial developments of ECCs date back to the 1990s, when research was directed at improving the cement matrix properties. The reinforcing fibrous phase, present at 2% by volume, has been shown to lend the subsequent cement matrix composite a degree of pseudo-ductility\(^1\) under stress, preventing failure in a brittle manner, which is more typical of cementitious mortars. More specifically, ECCs are able to undergo a process of multiple cracking under stress with the structure being preserved through stress transfer and a fibre-bridging mechanism: even at significant strain levels, crack widths are kept below 100 µm. In addition, at this level of reinforcement, the matrix of the fresh material remains workable during construction, an important feature of cementitious materials deployed on construction sites, particularly those where access is limited and the material must be pumped.

However, before the ECCs can be used in a large-scale commercial context, i.e. a tunnelling contract, there are a number of issues that must be addressed. These include: optimising material design and manufacturing routes (with reference to composition, fibre volume fraction, distribution and dispersion, and shrinkage behaviour in dry conditions representing the worst scenario), demonstrating that the pseudo-ductility can be achieved in large design geometries and understanding the long term durability of the ECC structure, with particular

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\(^1\) Pseudo-ductility for ECC is used to describe the plastic behaviour exhibited by the material under tensile stress and after the appearance of the initial crack. As the plasticity of the ECC material differs from the one exhibited by metals under stress for example, the term “pseudo” is added.
reference to the role of the fibre-matrix interface.

### 1.3 Aims and Objectives

The aim of the study is to contribute to the understanding of the issues related to the implementation of ECC materials in civil engineering projects, particularly as permanent linings in tunnelling applications (Figure 1-1).

![Diagram showing key issues of the implementation of ECCs](image)

Figure 1-1: Diagram showing key-issues of the implementation of ECCs

To demonstrate the wider potential of ECCs, three main areas need to be considered:

1. Mechanical performance: previous studies of ECC have mainly focussed on testing comparatively thin specimens in tension, typically 13 mm thick. In practice, however, ECCs are likely to be used in tunnel linings of thicknesses in the order of 50-150 mm (and potentially greater if their cost can be justified). Hence, the need to move from...
initial work based on thin specimens to thicker sections is essential if the behaviour of this material is to be understood more comprehensively, which will enable appropriate design criteria to be specified. When tested, ECCs are expected to exhibit pseudoplasticity under stress: this is evaluated in direct tensile testing with thick dog-bone geometry specimens and in flexure testing on beams. The tensile and flexural performance of the material is demonstrated along with a comparison with unreinforced cements. In order to have a better understanding of the parameters influencing the tensile behaviour, the effect of the casting media and specimen thickness are considered along with a fractographic analysis of the fractured surfaces of the specimens. Fibre dispersion and orientation are analysed and are linked with the tensile performance of the material, to make a correlation with the microstructure of the ECCs influenced by the “mixing” process and composition. In addition, single fibre pull-out testing from a cementitious matrix would give valuable information about the fibre-cement interface and would help with understanding of the conditions for pseudoplasticity of the material under stress. Finally, physical properties such as density and porosity of the specimens tested mechanically are measured and linked to the tensile and flexure test parameters to seek a link between the mechanical properties, the constituents of the ECCs and the manufacturing methodology. Such information will contribute to a better control of the mechanical properties of the ECCs for this application. Models of mechanical behaviour of composites in tension are applied to ECCs, which are verified and supplemented in order to have a better understanding of where ECCs fit with other materials and the novelty of the material.

2. Durability: the long-term durability is important for the application of the material in real life situations in civil engineering. The material should be able to exhibit pseudoplasticity under stress for its whole design life, which is likely to be of the order of 150 years, despite the possible degradation of the material over time and changes in the fibre-cement interfacial properties due to aging. A further design concept of the durability of the material is its ability to cope with damage (cracking) when it occurs, even if this is comparatively early in the structures’ life. To some extent, such durability may be achieved through a process known as self-healing of the cracks. The current work explores these areas.
Chapter 1 - Introduction

3. Shrinkage: shrinkage is associated with the formation of cracks and hence the risk of deterioration even as the structure is being produced. Furthermore cracks would also increase the permeability (to aggressive agents and any fluids) of the lining, which could be an issue for structures conveying water. As a consequence, before ECC can be used in a commercial context, it is important to understand and control shrinkage of the material. In the present work, both the effects of the environment and additives on shrinkage are considered. By understanding the key parameters that influence the shrinkage characteristics of ECC, it may then be possible to identify steps that can be taken to reduce or otherwise control the shrinkage behaviour of thick sections of the material used in engineering applications such as a tunnel lining.

The overall aim of the project is therefore to understand the link between the different parameters governing the ECC properties so as to optimise the ECC performance for the specific application of the material in tunnelling. Hence, the objectives of the current work are:

i. demonstrate the ability to cast large section test-pieces and the mechanical performance of test-pieces with greater thicknesses while understanding thickness effects on the mechanical performance;

ii. determine the physical properties of these test-pieces (i.e. density, porosity, fibre dispersion and orientation), understand the effects of these properties on mechanical performance and investigate how these may be controlled through materials selection and process control;

iii. determine the durability of the material;

iv. understand the (drying) shrinkage of such large specimens, and investigate methods for controlling this

All these objectives must be carried out in the light of the commercial context and the ability to use the material on-site: optimisation of the material includes a consideration of the financial implications of the material and its usability. In particular, a requirement is the ability to take a material that has been prepared carefully under laboratory conditions and from that, produce a form that can be manufactured in bulk quantities on-site under varying conditions and still give a reasonably consistent product that performs to its full potential.
1.4 Outline

This thesis consists of seven chapters. Following this Introduction, which has outlined the context within which the current work sits and the aims and objectives of this work, the second chapter presents a literature review. This review provides a perspective on the history of ECC, indicates the reasons for its suitability for civil engineering in general and tunnelling applications in particular and describes the current understanding with respect to the mechanical performance of ECCs in relation to their composition. Durability and shrinkage of the material are also considered.

Chapter 3 is concerned with the materials and methods used in the current work. The techniques used to assess the mechanical properties, the fibre matrix interface (in relation with the durability of the material), shrinkage and the physical properties (such as density and porosity) are detailed, together with a methodology to characterise fibre dispersion and orientation.

Chapters 4-6 present the results obtained from specific areas of research: mechanical properties, durability and shrinkage. Aspects relating to physical properties and autogenous healing will be included in the appropriate discussions arising from the test data.

The final chapter will present concluding remarks, providing a summary of the work, key findings and recommendations for future work.

Conference and journal papers arising from the research study are included as an Appendix.
2 Literature Review

2.1 Introduction
Reinforcing cementitious materials is not a new concept and fibres have been used as a reinforcement since ancient time, e.g. horsehair to reinforce mortars and straw in mud bricks (Jain and Kothari, 2012). Asbestos became very popular as a reinforcement material in cement and concrete in the late nineteenth century (Smith and Saunders, 2007); although, due to health risks presented by the material, a replacement needed to be found. In the mid-20th century, fibre reinforced composites became a class of material of increasing interest and different types of fibres have been used including glass, carbon, steel and polymeric fibres, and the research in this type of composite continues today.

In the first part of this chapter, the industrial application of ECCs, particularly in tunnel lining, is presented along with the required properties. Following this, fibre reinforced cement composites in general are described. Then, the principles of composites mechanics are discussed including the ACK theory. Finally, the required properties of the ECCs for its application in civil engineering such as pseudo-ductility, durability and shrinkage are explored with the parameters affecting these.

2.2 Industrial Context

2.2.1 Overview - Cements and Concretes
Cement material is often referred to as a binder, a fine powder which in contact with water, sets and hardens. Portland Cement (PC) is the most common construction material used in the World and in many respects the most durable (Naik, 2008). Cement is mainly used to produce concrete: a composite material made of cement, sand, coarse aggregates and water. Concrete, the most commonly used structural material (Neville, 2000), challenges most construction materials such as wood for example, as it does not require much in the way of maintenance or repair. Hence, structures made with concrete are often expected to be durable and last forever. Concrete, thanks to its excellent strength in compression, is the material of choice for most construction projects. However, concrete is brittle in tension, which can be problematic in situations where there is bending or a complex mix of loading modes. Therefore, in order to perform well in certain situations and structures, particularly
under tensile stresses, cementitious materials need to be reinforced.

### 2.2.2 Building with Fibre Reinforced Cementitious Materials

By reinforcing a cementitious material with fibres, a composite material with new and unique properties is created. Concrete, being weak in tension, is often reinforced with steel which is strong in tension, to offset this limitation. Further, cementitious materials, which are intrinsically brittle materials can under tensile stress exhibit enhanced mechanical properties when reinforced with a sufficient volume fraction of a fibrous phase.

The use of steel fibre reinforced concrete is now well established in the construction community (Concrete Society, 2007a) and can increase the ability to control crack widths if at sufficient dosage. However, like traditional steel reinforcement, steel fibres are prone to corrosion and because of the small diameter of the fibres, even mild corrosion can represent significant loss of cross-section, hence it becomes necessary to use more costly stainless steel fibres. There is, therefore, within the industry, an interest in alternative solutions that incorporate non-metallic (polymer) fibre systems (Concrete Society, 2007b).

In the mid-20th century, the development of synthetic polymer resins such as polyester and epoxy presented the possibility of synthetic polymeric fibres for the formulation of novel material solutions, including fibre reinforced composites. The latter became a class of material of increasing interest for cementitious materials, as the fibres are able to decrease the brittleness and enhance the properties of the matrix (Li, 2003 and Naaman, 1987). Due to the development of a range of materials, different types of synthetic fibres have been used as a reinforcing material (including glass, carbon, aramid and polypropylene) and research on fibre reinforced cementitious materials in general, continues today. By using synthetic (as opposed to natural) fibres, a better control of the material properties is possible as the fibres are manufactured and tailored for specific performances. Manufactured fibres exhibit greater uniformity and their properties can, to greater or less extent, be tailored for specific applications.

According to the Japanese Society of Civil Engineers (2008), High Performance Fibre

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2 Cementitious materials refer to any building material containing cement and to which water may be mixed to form a plastic paste, these include cements and mortars (the latter contain small aggregates or sand).
Reinforced Cement Composite (HPFRCC) is a material comprising a cement-based matrix and short reinforcing fibres. HPFRCC can be highly ‘ductile’, exhibiting multiple fine cracks and pseudo strain-hardening characteristics under uni-axial tensile stress. The term HPFRCC was first used by Naaman (1987) to describe composite materials with both high strength and toughness\(^3\)-ductility. In the literature, the behaviour of HPFRCCs and ECCs under tensile stress is often referred to as ‘strain-hardening’. However, in practice, strain-hardening is a process by which the microstructure of the material undergoes plastic deformation due to an applied strain. Such changes are often seen in metals when dislocations are introduced and entangled through cold-working processes. In cement matrix composites, such processes are not possible. Instead, pseudo-ductility is observed in HPFRCCs and ECCs through the ability of the fibre reinforcement to prevent catastrophic failure when the matrix begins to crack.

Recent work has demonstrated the potential of a particular family of such materials called Engineered Cement Composites (ECCs), a sub-set version of HPFRCC, a class of material developed for Civil Engineering applications and formed from a high cement content mortar matrix reinforced with synthetic fibres (Li, 2003). Whilst suitable for a number of applications, they represent a great opportunity for hydraulic tunnels, particularly those for water distribution systems as in addition of preventing the risk of corrosion, the use of fine polymeric instead of steel fibres promote the formation of very fine instead of large cracks, hence limiting the ingress of water within the structure and therefore their deterioration.

### 2.2.3 ECCs for Tunnel Linings: Ductility, Durability and Watertightness

A tunnel may be viewed as an underground passage which may be open to air (e.g. for the purposes of rail or road transport) or filled with fluid (e.g. for carrying water, a hydraulic tunnel). Currently, hydraulic tunnels are often constructed with steel reinforced concrete, which may degrade with time as the steel is prone to corrosion, particularly if the conditions of passivation of the steel are not present (Poursaeed and Hansson, 2007). Such deterioration often arises from corrosion of the steel reinforcement, which can occur within the concrete without the reinforcement being obviously exposed to the environment (CTI Consultants, 2004).

\(^3\)Toughness refers to the area below the tensile stress-strain curve: the higher the area, the higher the toughness.
ECCs, using polymeric fibres and being able to present a pseudo-ductility under tensile stress, represent an opportunity to make a paradigm shift in the way hydraulic tunnels are constructed. Through pseudo-ductile behaviour under tensile stress (discussed in more detail in section 2.5), structures produced from ECC are able to support multiple cracking of the cement matrix, giving the material enhanced strain capacity before damage localisation (a major crack occurring and widening at one single location) occurs.

ECCs can be used for all forms of tunnel linings (and could be considered for extending the life of existing tunnels) where the most important lining design parameter is the internal water pressure. ECCs have the potential to be used both for the construction of new tunnels and the repair/rehabilitation of existing structures (Psomas and Eddie, 2009; Figure 2-1).

- In the new construction, ECCs form the external lining. ECCs are designed to carry high internal pressures, reducing the need for maintenance or repair. The use of ECCs as a permanent lining will enable the construction of all types of tunnel that are more durable, including hydraulic tunnels. ECCs can be used in all forms of tunnel construction: cast-in-place, sprayed, pre-cast and extruded. For a traditional cast-in-place application for example, ECC material can be used on its own.
- In the repair of existing tunnels and in order to effect improvements following re-specified of existing tunnel structures, ECCs can be used as a lining. For shallow tunnels, ECCs could be used as a thin layer (up to 150 mm), conferring to the tunnel the ability to withstand a high internal pressure whilst being durable.

In both cases, such a layer of ECC will confer to the tunnel the ability to withstand a high internal pressure whilst being durable (Figure 2-1). Given that future operational requirements may demand the water to be pumped rapidly and distributed to other reservoirs using a high internal pressure (often up to 6 bar in the UK), the use of ECC can be seen to be of particular importance. Further, some water tunnels in the UK, for example, operate as gravity pipelines and would be unable to cope with this high internal pressure and hence, the use of a lining, such as one of ECC, is essential to the repurposing of existing assets. Potentially, such a fundamental change in material will make structures cheaper, safer to build and more durable, as well as providing improved longevity.
In the context of repairing structures, Figure 2-2 illustrates the difference in the interface when using concrete and ECCs for repair (Li and Li, 2006). A larger crack is visible (65 µm compared with 20 µm) when repairing concrete with concrete, attesting to the existence of high interfacial stresses causing delamination arising from drying shrinkage, whilst ECC – thanks to its pseudo-ductility under stress – shows no cracking or very limited crack widths. This illustrates the advantage of ECC material over concrete and the suitability of ECCs for repairing existing concrete structures.

Zhang et al. (2006) evaluated the flexural performance of a concrete specimen upon which an ECC layer was applied (on the tensile side). This could be representative of the case where ECC material is placed as an external lining and is submitted to tensile stresses as in Figure 2-1. The application of an ECC layer lead to an increase in flexural strength and ductility under stress compared with a typical concrete specimen and this improves with the thickness of the ECC layer. This shows the suitability of ECC as a repair material for tunnel structures; thanks to its pseudo-ductility, ECC can adapt and fit the existing material without any cracking, hence reducing the cost. Kamada and Li (2000) specify that a smooth surface is preferred rather than a rough surface in order to obtain a durable repaired structure, as the smooth surface results
in more desirable crack patterns and crack widths, which is in contradiction to a typical approach where concrete is used to repair concrete.

![Concrete/Concrete](image1.png) ![ECC/Concrete](image2.png)

*Figure 2-2: Comparison of interface delamination and crack opening of layered repair with concrete and ECC material (Li and Li, 2006)*

Having introduced ECC materials, particularly in the context of tunnel lining applications, the next section will consider the literature in relation with fibre reinforced cement composites considering cements in general, the fibre reinforcement and different classes of fibre reinforced cements and their characteristics.

### 2.3 Fibre Reinforced Cement Composites

#### 2.3.1 Introduction

Cement-based materials are typically brittle under tensile stress: the material fails suddenly as soon as the matrix cracks (Neville, 2000). With reinforced cements, a low volume fraction of discontinuous fibres is used to improve the mechanical performance of the matrix, particularly with respect to applied tensile stresses (Lange et al., 1996). Different classes of reinforced cement materials exist and are classified as a function of composition and mechanical performance (Li, 2002a). In the first part of this section, the characteristics of Portland Cement are presented with particular reference to its potential as a matrix. Secondly, fibres as a form of reinforcement are detailed with a particular focus on the resulting properties. In the third part, different classes of fibre reinforced cements are presented with their physical and mechanical characteristics.
2.3.2 Characteristics of Cements

Portland Cement (PC), named after its resemblance to a stone present on the Isle of Portland, is the most common type of cement used in concrete structures (Isaacs, 2008). In the late 60s, the European Economic Community (EEC) initiated the development of a standard for cement (Dhir and Jones, 1994). In the early 80s, a Technical Committee instructed by the European Committee for Standardisation (CEN) prepared a standard for PC (EN-197) to be used in Western European countries for plain and reinforced concrete. This lead to the development of BS EN 197-1 (Dhir and Jones, 1994).

Cement is made of two main constituents: clinker (the reactive component in cement) and gypsum. PC has the advantage of offering versatility, durability and adaptability to most applications. Furthermore, specific properties can be obtained by controlling the grain size and making specific additions to produce different cement types. For example, expansive cement can be obtained by mixing PC with clinkers of calcium sulfo-aluminate and gypsum, giving a cement that can, in some circumstances, compensate for the shrinkage observed during the life of a concrete structure (Mehta and Monteiro, 2006).

The manufacturing process of PC comprises several stages. Limestone CaCO$_3$ and clay Al$_2$O$_3$.2SiO$_2$.2H$_2$O are mixed together and ground into a powder. The mixture is then heated to approximately 1450 °C, whereupon partial fusion occurs. At 800 - 900 °C, limestone reacts to produce lime (CaO).

\[ CaCO_3 \rightarrow CaO + CO_2 \]  \hspace{1cm} (2.1)

Lime can then react with the clay components to form reactive oxides. The mixture is cooled rapidly to produce nodules or lumps of clinker of diameter of about 3-25 mm. The PC clinker is then mixed with gypsum (CaSO$_4$), a setting time controller, and ground to fine particles to produce cement. A typical PC clinker has approximately the following composition: 60-67 % CaO, 17-25 % SiO$_2$, 3-8 % Al$_2$O$_3$, 0.5-6 % Fe$_2$O$_3$ and 0.5-4 % other components (Neville, 2000). The four major oxides of importance to the cement industry are Alite, Belite, Aluminate and Ferrite (Table 2-1). Cement Chemist Notation (CCN) is usually used as a shorthand when describing these minerals: here oxides are referred to simply by the first letter of the element.
bonded to oxygen, as indicated in Table 2-1 (C = CaO; S = SiO$_2$; A = Al$_2$O$_3$; F = Fe$_2$O$_3$; H = H$_2$O).

As specified in the European Standard (EN 197-1), PC should consist of at least two-thirds by mass of calcium silicates (Alite and Belite), the remainder being aluminium and iron containing clinker phases and other compounds; the ratio by mass of CaO/SiO$_2$ shall be not less than 2.0 and the content of magnesium oxide (MgO) shall not exceed 5.0 % by mass (BS EN 197-1).

Table 2-1: Mineral phases present in a typical clinker (Taylor, 1997)

<table>
<thead>
<tr>
<th>Mineral phases</th>
<th>Cementitious appellation</th>
<th>Chemical composition</th>
<th>Cementitious notation</th>
<th>Content of the clinker (% weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tricalcium silicate</td>
<td>Alite</td>
<td>3CaO.SiO$_2$</td>
<td>C$_3$S</td>
<td>50-70 %</td>
</tr>
<tr>
<td>Dicalcium silicate</td>
<td>Belite</td>
<td>2CaO.SiO$_2$</td>
<td>C$_2$S</td>
<td>15-30 %</td>
</tr>
<tr>
<td>Tricalcium aluminate</td>
<td>Aluminate</td>
<td>3CaO.Al$_2$O$_3$</td>
<td>C$_3$A</td>
<td>5-10 %</td>
</tr>
<tr>
<td>Tetracalcium aluminoferrite</td>
<td>Ferrite</td>
<td>4CaO.Al$_2$O$_3$.Fe$_2$O$_3$</td>
<td>C$_4$AF</td>
<td>5-15 %</td>
</tr>
</tbody>
</table>

In contact with water, cement dissolves and produces ions (such as Ca$^{2+}$, Na$^+$, K$^+$, O$^{2-}$ and SO$_4^{2-}$, where $\overline{S}$ is the CCN for the latter) forming the pore solution. The silicates and aluminates of the PC (Table 2-1) react with water to form the products of hydration consisting of gel CaO.(SiO$_2$).H$_2$O (CSH and CH) and crystals of different structures such as Ettringite and Portlandite (Figure 2.3), producing the firm and hard hydrated cement paste (Neville, 2000).

Neville (2000) states that the silicates and aluminates listed in Table 2-1 have different rates of reaction: C$_4$AF is the most reactive followed by C$_3$A, C$_3$S and then C$_2$S. In the absence of gypsum, the reaction of C$_3$A is violent and could lead to the immediate stiffening of the cement paste. In the presence of gypsum (CaSO$_4$), C$_3$A reacts to form needles of Ettringite, mineral $\overline{C}_6$$\overline{A}$$\overline{S}_3$$\overline{H}_{32}$ (Figure 2-3a). Indeed, the dissolution stage is quickly followed by the formation of crystals of Ettringite; the reaction is exothermic and promotes the stiffness of the cement paste. In a limited amount of water, C$_3$S (as does C$_2$S) undergoes hydrolysis to produce a calcium silicate hydrate C$_3$S$_2$H$_3$ and some Ca(OH)$_2$. These products form the gel of the cement paste and the crystals of Portlandite (Ca(OH)$_2$): two-dimensional, hexagonal shaped crystals
(Figure 2-3b). These crystals, or plaques, form ‘stacks’ between the partially hydrated cement grains. However, there are some uncertainties as to whether C\textsubscript{3}S and C\textsubscript{2}S form the same calcium silicate products (Neville, 2000).

![Ettringite and Portlandite](image)

*Figure 2-3: Crystals found in a typical Portland Cement (a) Ettringite and (b) Portlandite (Baste et al., 2008)*

The microstructure of hydrated cement paste is complex, composed as it is of solids (Ettringite, calcium silicate hydrate (CSH), portlandite, monosulfate hydrate (C\textsubscript{4}A\textsubscript{2}S\textsubscript{3}H\textsubscript{12}) and residual un-hydrated cement), voids (air, capillary pores, inter-layer spaces) and water in different forms (capillary, adsorbed, interlayer and chemically combined). During the process of hydration, cement is placed in contact with water, where the hydration products such as CSH progressively form a shell around the un-hydrated cement as well as other products such as Ettringite and C-H, Figure 2-4 (Kurtis, 2007).

The water/cement ratio of the initial mixture is very important in the determination of the porous microstructure and controls many of the associated physical and mechanical properties of the hardened material. Additional parameters such as the characteristics of cement, chemical admixtures and the mixing procedures are also important. The hydration of PC in the presence of water takes place over time at a rate determined by the temperature and relative humidity. However, after a considerable amount of time, unhydrated cement particles remain in the hardened paste (Diamond, 2004). The presence of such unhydrated particles is potentially advantageous: if the cement paste cracks, for example due to shrinkage and/or mechanical load, in the presence of water, the hydration of unreacted cement can give rise to “autogenous” healing (Yang et al., 2009). This has implications for the durability of ECCs
Cement-based materials are brittle under tensile stress, due to their composition: different crystals, the microstructure consisting of different phases and the presence of significant porosity. Hariri-Ardebili and Mirzabozorg (2011) presented an idealised compressive and tensile stress-strain curve for concrete (Figure 2-5), showing a relatively high strength of the material in compression compared with a lower strength in tension. The behaviour of concrete when idealised is linear reaching its ultimate strength, after which it fails in a brittle manner. Cement paste, similarly to concrete, has a low fracture toughness resulting from its brittle failure in tension and has inherent defects within its structure. Furthermore, ettringite crystallising in the form of needles makes the structure more brittle and less durable, especially if its formation is delayed and does not occur in the plastic state of the hydration of the cementitious material (Stark and Bollmann, 1999).
2.3.3 Fibre reinforcement

The aim of incorporating fibres into cementitious matrices is to improve the properties of cement which is brittle, by making it pseudo-ductile under tensile stress. Reinforcing a material enables the creation of new properties that neither of the parent materials would have on their own. Different types of fibre are available for use to reinforce cement-based material such as paste, mortar or concrete. Table 2.2 presents different types of fibre with their respective physical characteristics and properties. The choice of fibres for a particular application depends on their characteristics and the ultimate use of the composite, but there will inevitably be a process of compromise. A range of fibres are available which offer high to very high strengths, however, ultimate strain should also be a consideration which will enable the material to be pseudo-ductile under stress. For example, steel fibres are often used due to their high stiffness (modulus of elasticity), but under some specific conditions depending on the exposure environment and crack widths, are prone to corrosion and present a relatively low ultimate strain capacity compared with other available fibres. The use of polymeric fibres in a cementitious matrix instead of steel fibres eliminates the risk of corrosion, although water and other factors can still induce deterioration of the fibres. Like steel fibres, Polyvinyl-alcohol (PVA) fibres offer a high tensile strength, as do aramid and carbon fibres. However, PVA fibres also have the advantage of presenting a high ultimate strain, important for achieving a high pseudo-ductility under stress.

Polymeric PVA fibres are likely to be, in this case, the reinforcement of choice. Concrete made of cement and relatively coarse aggregates allows a specific type of fibre such as steel to be effective as a reinforcing material for thicker sections of the composite thanks to the rigidity of the steel fibres. In comparison, cement mortar made of relatively fine particles (cement and sand) allows the use of thinner fibres to be very effective in reinforcing the cementitious matrix, while at the same time, ensuring the formation of thinner instead of wider cracks, as preferred for ECCs (Li, 2002a). This is probably because of the low dimensions of the fibres (present in a sufficient quantity), which are then able to bridge the very fine cracks, hence preventing these from increasing in width. This concept is detailed in section 2.3.4 where the crack width (crack opening) is determined as a function of the fibre characteristics.
Table 2-2: Properties of structural fibres

<table>
<thead>
<tr>
<th>Material</th>
<th>diameter (μm)</th>
<th>Unit Weight (t/m$^3$)</th>
<th>Tensile Strength (GPa)</th>
<th>Modulus of Elasticity (GPa)</th>
<th>Ultimate Strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel</td>
<td>5-500</td>
<td>7.85</td>
<td>0.5-2.0</td>
<td>200-210</td>
<td>0.5-3.5</td>
</tr>
<tr>
<td>Glass</td>
<td>9-15</td>
<td>2.62</td>
<td>2.0-4.0</td>
<td>70-80</td>
<td>2.0-3.5</td>
</tr>
<tr>
<td>PP</td>
<td>20-400</td>
<td>0.93</td>
<td>0.4-0.8</td>
<td>3.5-10</td>
<td>15.0-25.0</td>
</tr>
<tr>
<td>Aramid</td>
<td>10-12</td>
<td>1.45</td>
<td>2.3-3.5</td>
<td>63-120</td>
<td>2.0-4.5</td>
</tr>
<tr>
<td>PVA</td>
<td>25-40</td>
<td>1.31</td>
<td>1.2-1.6</td>
<td>25-40</td>
<td>7.0-8.0</td>
</tr>
<tr>
<td>Carbon</td>
<td>23-400</td>
<td>1.65</td>
<td>2.5-4.0</td>
<td>230-380</td>
<td>0.5-1.5</td>
</tr>
</tbody>
</table>

2.3.4 Different classes of fibre reinforced cements

According to Li (2002a), cementitious materials reinforced with fibres may be divided into three groups, depending on their composition and behaviour under tensile stress (Table 2-3). The first group, fibre reinforced composites (FRCs), typically use a low fibre (usually steel) content of the order of 2 vol. % and exhibit a strain-softening behaviour$^5$. The second group, high performance fibre reinforced composites (HPFRCs) have the ability to present pseudo-ductile behaviour$^6$ under tensile stress. HPFRCs using much larger volumes of fibres than FRCs ($V_f > 5$ vol. %) present limited pseudo-ductile behaviour with a tensile strain capacity of the order of 1.5 %. The last group, engineered cement composites (ECCs), represent an improvement in the properties of HPFRCs because they use a lower volume fraction of fine fibres whilst exhibiting a higher tensile strain capacity thanks to the successive transfer of the load from the fibre to the matrix via the fibre/cement interface for the formation of multiple and finer cracks. ECCs have the ability to exhibit pseudo-ductile behaviour under stress thanks to the incorporation of very fine tailored (for the cementitious matrix) polymeric fibres leading to a large number of cracks with crack widths not exceeding 100 μm, instead of a smaller number of cracks with larger crack widths. ECCs are therefore considered as an improved version of HPFRC materials. These materials have a typical polymeric fibre content of 2 vol. %

---

$^4$ Structural fibres refer to fibres which can be used to build structures such as bridges, tunnels for example.

$^5$ Strain-softening behaviour under stress is characterised by a peak of stress at a specific strain corresponding the formation of the crack, followed by a slow decrease in the stress, by the fibres bridging the existing crack.

$^6$ In the literature, this is often referred to as 'strain-hardening'. In practice, strain-hardening is a process by which the microstructure of the material undergoes plastic deformation due to an applied strain. Such changes are often seen in metals when dislocations are introduced and entangled through cold-working processes. In cement matrix composites, such processes are not possible. Instead, pseudo-ductility is observed in HPFRCs and ECCs through the ability of the fibre reinforcement to prevent catastrophic failure when the matrix begins to crack.
and exhibit a pseudo-ductile behaviour under stress with ultimate strains in the range of 3-8% being reported (e.g. Li, 1998; Li et al., 2001). It should be noted that Li’s work on ECC focussed on PVA fibres and aimed at improving the matrix properties. It showed that 2 vol. % is the ideal loading: below 1.5 %, multiple cracking associated with the pseudo-ductility under stress cannot occur, whilst a higher percentage of fibres (above 2.5 %) reduces the workability and increases the cost (Li, 2002b), which is a specific limit for a given set of assumed parameters.

The differences in mechanical properties between FRC and HPFRC (or High Performance Fibre Reinforced Cement-based Composites, HPFRCC) often assimilated as ECC in the literature, are presented in Figure 2-6 (Kuder and Shah, 2010).

Table 2-3: Comparison between FRC, HPFRC and ECC (Li, 2002a)

<table>
<thead>
<tr>
<th></th>
<th>FRC</th>
<th>HPFRC</th>
<th>ECC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composite Design Methodology</td>
<td>NA</td>
<td>Use high ( V_f )</td>
<td>Micromechanics based, minimise ( V_f ) for cost and processability</td>
</tr>
<tr>
<td>Fibre</td>
<td>Any type/ ( V_f ) usually &lt; 2 %; ( d_f ) (steel) ( \sim ) 500 ( \mu )m</td>
<td>Mostly steel/ ( V_f ) usually &gt; 5%, ( d_f ) ( \sim ) 150 ( \mu )m</td>
<td>Tailored, polymer fibres most suitable; ( V_f ) usually &lt; 2%; ( d_f ) &lt; 50 ( \mu )m</td>
</tr>
<tr>
<td>Matrix</td>
<td>Coarse aggregates used</td>
<td>Fine aggregates used</td>
<td>Controlled for matrix toughness and initial flaw size; fine sand used</td>
</tr>
<tr>
<td>Interface</td>
<td>Not controlled</td>
<td>Not controlled</td>
<td>( G_d ) and ( \tau_d ) controlled</td>
</tr>
<tr>
<td>Tensile behaviour</td>
<td>Strain-softening</td>
<td>Strain-hardening</td>
<td>Strain-hardening</td>
</tr>
<tr>
<td>Tensile strain capacity</td>
<td>0.1 %</td>
<td>&lt; 1.5 %</td>
<td>&gt; 3 %; 8 % demonstrated</td>
</tr>
<tr>
<td>Crack width</td>
<td>Unlimited</td>
<td>Typically several hundred ( \mu )m, unlimited for ( \varepsilon &gt; 1.5% )</td>
<td>Typically &lt; 100 ( \mu )m during strain-hardening</td>
</tr>
<tr>
<td>Processing</td>
<td>Self-compaction demonstrated; Extrudability demonstrated</td>
<td>Self-compaction impossible due to high ( V_f ), often requires high frequency vibration (e.g. in CRC); Extrudability demonstrated</td>
<td>Self-compaction demonstrated; Extrudability demonstrated</td>
</tr>
</tbody>
</table>

Workability is defined as the ease of placement and the resistance to segregation of concrete (Neville, 2000) and in general, to its ability to deform in its fresh state. The term has become obsolete with the introduction of consistency (BS EN 206: 2013).
The theoretical model presenting pseudo-ductile behaviour under tensile stress has been developed by Naaman (1987), who was the first to mention the concept of high performance fibre reinforced cementitious composites. Figure 2-7 compares the strain-softening behaviour of a conventional FRC material under stress with a strain-hardening behaviour of an HPFRCC material. For an HPFRCC (or ECC in our context) material, the stress strain curve presents an elastic region up to the first crack, followed by a pseudo-ductile region where multiple-cracking occur, characterised by stress and strain coordinates. The peak point at the end of the curve corresponds to the maximum post-cracking stress $\sigma_{pc}$ and strain $\varepsilon_{pc}$. This concept of stress increasing after first cracking is further mentioned in section 2.4.3 when Naaman notes the critical volume fraction of the fibres $V_{f,\text{crit}}$ and the conditions to achieve a pseudo-ductile behaviour under tensile stress.
Having introduced fibre reinforced cement composites, the next section will present the principles of composite mechanics, hence enabling a better understanding and appreciation of the mechanical properties of the ECCs, and how these may be modelled and predicted.

### 2.4 Principles of Composite mechanics for fibre reinforced cements

#### 2.4.1 Introduction

In order to use the fibre reinforced cementitious material in civil engineering applications, the mechanical behaviour needs to be understood and appropriate design methods developed. This section provides an overview of some key aspects, particularly the composite mechanics underlying the behaviour of ECCs under load, and shows how some well-established models used with composites can be applied in the current context. The theory behind the micromechanics used to design ECC by Li and co-workers is also detailed.
2.4.2 Rule of mixtures

Any composite material is based on combining at least two constituent materials, usually a reinforcement and a matrix. The addition of the reinforcement modifies the matrix properties in a way that depends upon the material properties and the volume fraction. The rule of mixtures is a simple way of determining the properties of a composite as a result of the quantity of each constituent material present in the composite and the corresponding material property. The density of a composite material is a good example of a property that follows a rule-of-mixtures prediction.

The rule-of-mixtures can also be applied to prediction of certain mechanical and physical properties along the fibre direction of a unidirectionally reinforced continuous fibre composite. Fundamental to understanding the mechanical properties, is the basic load-sharing between the fibre and matrix (Hull and Clyne 1996), which enables the mechanical stress applied to the composite in the fibre direction, $\sigma_c$, to be related to the corresponding stresses carried by the fibres, $\sigma_f$, and matrix, $\sigma_m$, and their volume fractions according to:

$$V_f \sigma_f + (1 - V_f) \sigma_m = \sigma_c$$  \hspace{1cm} (2.2)

where $V_f$ is the volume fraction of the reinforcement.

This equation leads to a simple expression for Young’s modulus, which is an important parameter for design, although modification is needed to deal with short oriented fibres. This is discussed in section 2.4.5, which details the ACK theory.

Equation 2.2 is based on assumptions that the fibre and matrix are able to deform independently of each other and remain elastic – under these conditions (provided the response of the composite is elastic), the proportion of the imposed load carried by the fibres and the matrix remain independent of the applied load. Hence, care should be taken when using the rule of mixtures for an expected elastic-plastic response of the composite under stress. In the next section, this is used as a basis for understanding the strength of brittle matrix composites.
2.4.3 Critical volume fraction of fibres \( (V_{f,\text{crit}}) \)

In composite systems where the matrix fails first, it is possible to identify a minimum necessary volume fraction of reinforcing fibre, so that the fibres are able to take the redistributed load when the matrix fails, without the fibres failing. If this requirement is met, then multiple cracking of the matrix and “pseudo-ductile” behaviour become possible. The minimum fibre volume fraction required to achieve this condition is termed the critical fibre volume fraction, \( V_{f,\text{crit}} \).

In the situation where the matrix has failed, the ultimate strength of the composite is determined by the fibre strength, \( \sigma_{fu} \), and is given by:

\[
\sigma_{cu} = V_f \sigma_{fu}
\]  

Equation 2.2 can be used to determine the stress on the composite just before matrix failure occurs. Hence, Naaman and Reinhardt (1996), define the condition for an aligned fibre composite material to exhibit pseudo-ductile behaviour with multiple cracks under stress, as:

\[
\sigma_{fu} V_f \geq \sigma_{mu} (1 - V_f) + \epsilon_{mu} E_f V_f
\]

where \( \sigma_{fu} \) is the ultimate tensile strength of the fibre, \( V_m, E_m \) and \( V_f, E_f \) being the matrix and fibre volume fraction and Young’s modulus respectively, \( \sigma_{mu} \) and \( \epsilon_{mu} \) are the ultimate matrix strength and strain respectively.

Equation 2.4 describes the condition for the fibres which are able to carry the load after initial matrix failure by comparing the composite strength capacity post-cracking with the stress level at which first cracking occurs. If this is met, then after initial matrix failure, the load will be transferred to another area of the matrix for further cracking: a process known as multiple-cracking (Bentur and Mindess, 2007).

Equations 2.3 and 2.4 are plotted schematically in Figure 2.8. Aveston et al. (1971) consider the intersection of curves (1) and (2) as determining the critical volume fraction of fibres \( V_{f,\text{crit}} \) for ductile fibre/brittle matrix composite material.
The re-arranging of equation 2.4 gives:

\[ \sigma_{mu} = V_{f, crit} (\sigma_{fu} + \sigma_{mu} - \epsilon_{mu}E_f) \]  

(2.5)

Hence, the following condition can be written:

\[ V_{f, crit} = \frac{\sigma_{mu}}{\sigma_{fu} + \sigma_{mu} - \epsilon_{mu}E_f} \]  

(2.6)

The simplification of the previous relation assuming \( E_c \approx E_m \), leads to equation 2.7:

\[ V_{f, crit} \geq \frac{\sigma_{mu}}{\sigma_{fu}} \]  

(2.7)
The strength of the fibres will contribute to the strength of the composite only when $V_f > V_{f,\text{crit}}$ (Bentur and Mindess, 2007). Hence the importance of $V_{f,\text{crit}}$.

Equation 2.7 should be considered as a lower estimation of $V_{f,\text{crit}}$ as additional factors need to be taken into account when considering the failure stress such as flaw size and matrix fracture energy, which could be determined with a wedge splitting test (Wittmann, 2002). The importance of exceeding $V_{f,\text{crit}}$ is demonstrated in a study on fibre reinforced brittle matrix composites (Li and Leung, 1992), where $V_{f,\text{crit}}$ was computed to be 0.3 %. Using a volume of fibres of 0.1 % shows a catastrophic failure, whereas when a fibre content of 1 % is used, the composite shows significant pseudo-ductility under stress. In using such an approach with cementitious matrices, it is also important to remember that such materials continue to cure, harden and gain in strength throughout their lifetime and hence $V_{f,\text{crit}}$ should, to some extent, consider future performance. Furthermore, equation 2.7 assumes aligned fibres whereas for non-oriented short fibres composites, the minimal volume of fibres can be higher than those predicted with the previous equation (Naaman and Reinhardt, 1996). Hence, the condition in equation 2.7 is not valid for discontinuous, randomly distributed fibres, where further calculations are necessary to account for the reduced contribution of non-aligned fibres.

Bentur and Mindess (2007) consider that for short fibre composites, failure of the material in the post-cracking zone occurs primarily by fibre pull-out, hence equation 2.4 can be adapted to account for fibre pull-out, by replacing the average fibre stress at fracture by the resistance of the fibre to pull-out assuming a constant frictional bond strength. This gives equation 2.8:

$$\sigma_{cu} = \eta_{\theta} V_f \tau_0 \frac{L_f}{d_f}$$

(2.8)

where $\eta_{\theta}$ is the fibre orientation distribution factor (defined in more detail in section 2.4.4), $\tau_0$ is the frictional bond strength, $L_f$ and $d_f$ are the length and diameter of the fibre respectively.

Hence, using equation 2.7, $V_{f,\text{crit}}$ becomes:

$$V_{f,\text{crit}} = \frac{\sigma_{mu}}{\eta_{\theta} \tau_0} \frac{1}{L_f/d_f}$$

(2.9)

---

8 Flaw could be defined as a defect in the structure of the material.
Naaman (1987) described the condition to achieve a pseudo-ductile behaviour stating the post-cracking strength of the composite must be higher or equal to the strength at first cracking of the composite. This is presented as:

\[ \sigma_{pc} \geq \sigma_{cc} \]  \hspace{1cm} (2.10)

where \( \sigma_{pc} \) is the post-cracking strength or the maximum stress in the composite after first cracking and \( \sigma_{cc} \) is the strength at first cracking of the composite. The terms \( \sigma_{pc} \) and \( \sigma_{cc} \) could be defined as:

\[ \sigma_{cc} = \sigma_{mu}(1 - V_f) + \alpha\tau_0 V_f \frac{L_f}{d_f} \]  \hspace{1cm} (2.11)

\[ \sigma_{pc} = \lambda\tau_0 \frac{L_f}{d_f} V_f \]  \hspace{1cm} (2.12)

where \( \sigma_{mu} \) is the ultimate tensile strength of the matrix, \( \alpha \) and \( \lambda \) are coefficients which are a product of several other coefficients that depend on variables such as fibre distribution, orientation and bond efficiency; \( L_f \) and \( d_f \) are the length and the diameter of the fibres respectively and \( \tau_0 \) is the average frictional bond strength at the fibre-cement interface.

Hence, substituting equations 2.11 and 2.12 into equation 2.10, the critical volume fraction of fibre \( V_{f,\text{crit}} \) necessary to obtain a pseudo-ductile behaviour under tensile stress can be determined:

\[ V_f \geq \frac{1}{1 + \frac{\tau_0}{\sigma_{mu}d_f} \frac{L_f}{\lambda - \alpha}} = V_{f,\text{crit}} \]  \hspace{1cm} (2.13)

Equation 2.13 can also be written as:

\[ V_f \frac{\tau_0 L_f}{\sigma_{mu}d_f} \geq \frac{\lambda - \alpha}{1 - V_f} \]  \hspace{1cm} (2.14)
Because $V_f$ is relatively small, therefore equation 2.14 becomes

$$V_f \frac{\tau_0 \, L_f}{\sigma_{mu} \, d_f} \approx \frac{1}{\lambda - \alpha} \quad (2.15)$$

Hence, $V_{f,\text{crit}}$ could be given by the following relation:

$$V_{f,\text{crit}} = \frac{\sigma_{mu} \, d_f}{\tau_0 \, L_f} \frac{1}{\lambda - \alpha} \quad (2.16)$$

Equation 2.16, similarly to equation 2.9 illustrates the influence of several parameters on the mechanical performance of the material, including the aspect ratio of the fibre and the frictional bond at the fibre-matrix interface. However, compared with the previous expression of $V_{f,\text{crit}}$, equation 2.16 does not depend upon the fibre strength, because the strength is assumed to be controlled by fibre pull-out.

Based on an energy approach developed by Marshall and Cox (1988)\textsuperscript{9}, Li (1997) indicates the condition to obtain a pseudo-ductile behaviour associated with a steady-state cracking occurring (concept developed in section 2.4.6 and associated with the pseudo-ductile behaviour of ECC) as the steady-state cracking stress $\sigma_{ss}$ which must be less than the maximum bridging stress $\sigma_0$:

$$\sigma_{ss} \leq \sigma_0 \quad (2.17)$$

Based on equation 2.17, Li expressed in equation 2.18 a critical fibre volume fraction for typical ECCs above which the composite will show pseudo-ductility:

$$V_{f,\text{crit}} = \frac{12 J_c}{g \tau_0 (L_f/d_f) \delta_o} \quad (2.18)$$

where $J_c$ is the crack tip toughness or can be approximated as the cementitious matrix

\textsuperscript{9} Based on a J-integral analysis of a steady state crack, where $J_c$ refers to the crack tip toughness. For steady state cracking to occur, which ensures multiple cracking of the matrix under stress, the steady state cracking stress must be less than the maximum bridging stress in the bridging law.
toughness in most fibre reinforced cementitious composites with less than 5 % fibre volume fraction (Li, 1997), g is the snubbing factor taken as equal to 2, Lf is the length of the fibre, df the diameter of the fibre, τ₀ is the fibre/matrix frictional bond strength and δ₀ is the crack opening corresponding to the maximum bridging stress.

Equation 2.18 is based on the steady state crack analysis and depends on the crack tip toughness, crack opening, fibre cement interface and physical aspects of the fibre.

The crack opening corresponding to the maximum bridging stress δ₀ can be calculated using equation 2.19:

\[
\delta_o = \frac{\tau_0 L_f^2}{E_f d_f (1+\eta)}
\]  

(2.19)

where \( E_f \) is the Young’s modulus of the fibres.

The \( \eta \) term can be calculated using equation 2.20:

\[
\eta = \frac{V_f E_f}{V_m E_m}
\]  

(2.20)

The analysis of the literature (Li, 1997) reveals a relation between \( V_{f,crit} \) and the fibre-cement bond strength for different values of crack tip toughness. For example, when \( J_c \) equals 0.015 kJ.m⁻² and for a bond strength of 0.8 MPa, \( V_{f,crit} \) shows a value of 1.3 % as calculated for the system detailed in Li’s study. Li (1997) establishes the evaluation of \( V_{f,crit} \) for ECCs and hence the influence of parameters specific to ECCs is expected to be taken into consideration. Therefore, it is believed that the latest method would be more appropriate for our current study. However, the fact that this does not take into consideration the fibre strength could suggest that the method developed by Aveston et al. (1971) might be more suitable. These relations will be evaluated in chapter 4.

**2.4.4 Classical short-fibre composite theory and fibre orientation**

Thick ECCs present considerable challenges in terms of developing predictive models for mechanical performance. Given the unresolved issues around modelling the development of
damage and failure in continuous fibre laminated composites (Kaddour et al., 2013), it is clear that ECCs present further levels of complexity in terms of the nature of the reinforcement – a low volume fraction of partially aligned short fibre and the uncertain role of matrix defects. Aspects of multiple cracking behaviour may be amenable to modified versions of the classic ACK type of approach, as has been explored to some extent in papers by Li and co-workers (Li and Leung, 1992) for less thick (and with correspondingly better fibre alignment) ECCs than those which will be tested in the present work.

In the present section we assess simple strength models based first on fibre fracture and secondly on fibre pull-out.

Based on classical short fibre composite theory, the composite tensile stress is related to the fibre stress ($\sigma_f$), the matrix stress ($\sigma_m$) and the corresponding volume fractions ($V_f$ and $V_m$) according to:

$$\sigma_c = \eta_l \eta_\theta V_f \sigma_f + V_m \sigma_m$$  \hspace{1cm} (2.21)

The fibre length distribution factor ($\eta_l$) can be estimated using the Cox equation (Cox, 1952), which is based on the assumption of elastic transfer between the fibre and the matrix:

$$\eta_l = 1 - \left[ \tanh \left( \frac{\beta L_f}{2} \right) \right] / \left( \frac{\beta L_f}{2} \right)$$  \hspace{1cm} (2.22)

where $\beta$ is the shear-lag parameter (characterising the stress transfer between the fibre and the matrix in composites) and is defined by equation 2.23:

$$\beta = \frac{2\pi G_m}{E_f A_f \ln \left( \frac{R}{R_f} \right)}$$  \hspace{1cm} (2.23)

where $L_f$, $G_m$, $A_f$, $R_f$ and $R$ are the fibre length, the shear modulus of the matrix, the cross-sectional area of the fibre, the radius of the fibre and the mean separation of the fibre.

The fibre orientation distribution factor $\eta_\theta$ can be calculated using the Krenchel equation
(Krenchel, 1964):

\[ \eta_\theta = \sum_{i=0}^{i=180^\circ} V_i \cos^4 \theta_i \]  
(2.24)

In order for the cement composite to show multiple cracking and hence pseudo-ductility, the fibres have to be able to support the load on the composite, after initial matrix failure. In the case of fibre fracture, therefore the composite strength can be estimated from the fibre ultimate strength (\(\sigma_{fu}\)) according to:

\[ \sigma_{c,\text{max}} = \eta_l \eta_\theta V_f \sigma_{fu} \]  
(2.25)

This theory suggests a link between the fibre orientation and the composite mechanical performance. This is in good agreement with a study conducted on polypropylene fibres in a cementitious matrix (Takashima et al., 2003). It was observed that extruded specimens with approximately 80% of the fibre content aligned with respect to the extrusion direction exhibit enhanced mechanical performance compared with cast specimens having a broader distribution of fibre orientations. This finding may be of significance when dealing with ECC specimens manufactured with larger sections, than typically produced for laboratory studies.

In the case of fibre pull-out controlling failure and assuming that the fibre-matrix interface can be characterised by a constant fibre/matrix frictional bond strength \(\tau_0\), then the load to pull-out a single (aligned) fibre, \(P\), is simply given by:

\[ P = \pi d_f \bar{l}_{po} \tau_0 \]  
(2.26)

where \(\bar{l}_{po}\) is the mean fibre pull-out length and \(d_f\) is the fibre diameter.

Hence, the corresponding stress on the composite to initiate pull-out failure can be estimated from equation 2.27:

\[ \sigma_{po} = V_f \frac{P}{\pi R_f^2} = \frac{4V_f \tau_0 \bar{l}_{po}}{d_f} \]  
(2.27)
2.4.5 ACK Theory

Aveston et al. (1971, 1973) were the first to attempt to model the behaviour of a brittle matrix composite by developing an energetics-based (fracture mechanics) model for the matrix failure process (Cuypers et al., 2004). This is known as “ACK Theory”. The ACK approach considers the existence of three zones in a typical stress-strain curve of a brittle matrix composite as illustrated in a study on textile reinforced composites (Figure 2-9). The three zones are the pre-cracking zone, the multiple cracking zone and finally the post-cracking zone. Blom et al. (2008) illustrate this concept in Figure 2-9.

![Figure 2-9: Typical experimental and theoretical stress-strain curve, according to the ACK theory (Blom et al., 2008)](image)

- **Pre-cracking zone (Zone I)**
  The pre-cracking zone, or zone I, corresponds to a linear elastic region where the Young’s modulus of the composite can be estimated from the rule of mixtures as:

  \[ E_c = E_f V_f + E_m V_m \]  

  (2.28)

  where the matrix volume fraction \( V_m \) and the fibre volume fraction \( V_f \) are related by:

  \[ V_m = 1 - V_f \]  

  (2.29)
The terms $E$ and $V$ represent the Young’s modulus and volume fraction respectively, whilst the subscripts $f$ and $m$ signify the fibre and the matrix respectively.

However, equation 2.28 is based on continuous aligned fibres, whereas ECCs contain discontinuous and randomly distributed fibres. Hence, the need to introduce equations 2.30 and 2.31:

$$E_c' = E_f V_f^* + E_m V_m$$

(2.30)

The volume of fibres $V_f^*$ should take into consideration the fibre length distribution factor ($\eta_l$) and the fibre orientation distribution factor ($\eta_\theta$).

$$V_f^* = \eta_l \eta_\theta V_f$$

(2.31)

where $\eta_l$ and $\eta_\theta$ are defined in section 2.4.4.

- **Multiple-cracking zone (Zone II)**

Zone II corresponds to a region of the curve where multiple cracking takes place. The first crack occurs typically at relatively low tensile stress/strain levels (compared to the strength of the fibres), as the tensile strength of the brittle matrix is quite low. ACK theory (Mileiko, 1997) gives an expression of the strain at which the first matrix crack occurs $\varepsilon_{muc}$ as:

$$\varepsilon_{muc} = \left( \frac{12 \tau_0 \gamma_m E_f V_f^2}{E_c E_m^2 R_f V_m} \right)^{\frac{1}{3}}$$

(2.32)

where $E_f$, $E_m$ and $E_c$ are the Young’s moduli of the fibre, matrix and composite respectively; $V_f$ and $V_m$ are the volume fraction of the fibre and matrix respectively; $R_f$ is the fibre radius; $\gamma_m$ is the matrix fracture energy. Li (1997) mentions that the crack tip toughness $J_c$ can be approximated as $\Upsilon_m$ in most fibre reinforced cementitious composites with less than 5 % fibre volume fraction and $\tau_0$ is the fibre/matrix frictional bond strength. The equation demonstrates that although first cracking is a matrix event, it depends on various fibre and composite parameters.
As ACK has been developed for long and aligned fibres, a simple modification of equation 2.32 would be to introduce \( V_f^* \) to take into consideration fibre orientation (this will be considered further in chapter 4). However, as cited by Bentur and Mindness (2007), Tjiptobroto and Hansen (1993) extended the ACK theory to short and oriented fibre composites: equations 2.33 and 2.35.

The first equation is based on the assumption that the stress transfer length is equal to the fibre length \( L_f \) and that there is a linear strain distribution along the fibre:

\[
\varepsilon_{muc} = \left( \frac{2\gamma_mv_m}{\left[(3/4)E_c-(7/24)E_fV_f^*(1+\alpha)L_f\right]} \right)^{1/2}
\]  

(2.33)

Where

\[
\alpha = \frac{E_mV_m}{E_fV_f}
\]

(2.34)

The second equation assumes that the stress transfer length is smaller than the fibre length and again that there is a linear strain distribution along the fibre:

\[
\varepsilon_{muc} = \left( \frac{2\gamma_mv_m}{\left[(3/4)E_c-(7/24)E_fV_f^*(1+\alpha)\alpha(\beta L_f)\right]} \right)^{1/2}
\]

(2.35)

where:

\[
\beta = \frac{L_{tr}}{L_f/2}
\]

(2.36)

and:

\[
L_{tr} = \frac{d_f\varepsilon_{mu}(1+\alpha)E_f}{4\tau_0}
\]

(2.37)

Once the matrix cracks and transfers the load to the fibres with the associated debonding of the matrix-fibre interface (as it is assumed that the matrix-fibre bond is weak), the theory stipulates that along the debonded interface, there is a constant frictional matrix-fibre interface shear stress. Away from the plane of the matrix crack, the interface shear stress
enables stress transfer from the fibres to the matrix. At a distance from the crack face, the matrix stress reaches a maximum value $\sigma^f_m$ expressed as:

$$\sigma^f_m = \frac{E_m \sigma_c}{E_c}$$  \hspace{1cm} (2.38)

where $\sigma_c$ is the applied composite stress.

A study carried out on single fibre/bismaleide composites (Park et al., 2002) illustrated the concept of the ACK theory. Once a crack appears in the matrix, further cracking cannot occur in the same area as the region close to the crack is relieved of stress. However, the strain in the matrix increases with the distance from the crack at a rate determined by the maximum shear stress that can be developed by the interface until reaching failure strain at a distance $x$, also called transfer length, away from the crack where cracking of the matrix could occur at cracking stress. The transfer length at which cracking stress is reached within the ACK framework is given by:

$$x = \frac{V_m \sigma_{mu} R_f}{2 V_f \tau_o}$$  \hspace{1cm} (2.39)

where $V_m$ is the volume fraction of the matrix, $\sigma_{mu}$ is the ultimate matrix stress and can be taken here as stress at which the crack begins, $R_f$ is the radius of the fibre and $V_f$ the volume fraction of fibre.

The ACK model is designed for continuous and aligned fibres. In order to adapt it to ECC, equation 2.39 can be modified through the fibre volume fraction, which accounts for fibre orientation:

$$x^* = \frac{V_m \sigma_{mu} R_f}{2 V_f^* \tau_o}$$  \hspace{1cm} (2.40)

Whilst equation 2.39 is valuable for continuous aligned fibres, its modified version (equation 2.40) would be more appropriate for discontinuous random fibres. An alternative
modification, which incorporates the fibre length explicitly, is given by Wu and Li (1995) as expressed in equation 2.41:

\[ x_d = \frac{L_f - \sqrt{L_f^2 - 2\pi L_f \phi x}}{2} \]  

(2.41)

where \( \phi = \frac{4}{\pi g} \)  

(2.42)

where \( g \) is the snubbing factor taken as equal to 2.

From these values and knowing that the saturation crack spacing occurs at between one and two times the transfer length (and that the saturation crack spacing is never bigger than twice the transfer length), then the mean crack spacing can be estimated as \( x_f \), equation 2.43:

\[ x_f = \frac{3}{2} x \]  

(2.43)

Although ACK specifies a difference between a distance \( x \) at which a crack can occur (called the transfer length) and the mean crack spacing at which cracks occur, it is interesting to note that some researchers such as Wu and Li (1995) assume that \( x \) is the crack spacing suggesting that as soon as the cracking stress is reached then, cracking occurs. This will be investigated further in chapter 4.

- **Post-cracking zone (Zone III)**

Zone III, the post cracking zone, starts when all the multiple cracking in the composite matrix have occurred. The material is then held together by the fibres only until complete failure of the composite. The composite stiffness is therefore a function of fibres only:

\[ E_{c_{III}} = E_f V_f^* \]  

(2.44)

However, ACK theory has a number of limitations. First, the ACK theory assumes that the fibre-matrix bond is weak promoting immediate de-bonding of the matrix-fibre interface under stress, whereas depending on the material used to make the composite, the fibre-matrix bond
can be very strong, especially when a chemical bond is present. Second, the ACK theory also assumes that the frictional bond strength $\tau_0$ is constant with the slip at the de-bonded matrix-fibre interface, whereas this assumption could depend on the fibre surface coating (such as the presence of an oiling agent on the fibre surface) and also the presence of the coating only on specific areas of the fibres, creating different frictional stresses with the slip under stress. Harris et al. (1992) looking at fibre bridging in GFRP composites stipulate that the matrix cracking stress can be predicted by the ACK theory; however subcritical micro-cracks can form at stresses below the predicted critical values without affecting the composite properties. In addition, in the ACK theory, it is considered that all matrix cracking occurs at the same stress level and that fibres are held only by frictional stresses. These latest assumptions have therefore to be carefully considered, particularly in composite materials involving different types of interaction between the fibre and matrix. Fibre debonding and sliding can have an effect on the fracture behaviour of fibre-reinforced composites, such that the interaction between fibre and matrix can influence the toughness of the (brittle) matrix (Zhang et al., 2004).

2.4.6 Micromechanics for ECC Design

2.4.6.1 Introduction

Significant research and development in ECC has been carried out to understand how the microstructure and material optimisation can lead to a greater ductility. Materials selection also plays an important role in reducing the cost especially as PVA fibres are relatively expensive. Li (2002b) used a micromechanics approach to relate the macroscopic properties to the microstructure of the composite. A summary of the relevance of this in the current work is presented in this section.

2.4.6.2 Fibre bridging properties

Li (2002b), studying ECCs, demonstrated the link between the micromechanical parameters (fibre, matrix and interface) and the pseudo-ductile behaviour of the resulting composite under stress.

Figure 2-10 shows that the pseudo-ductile behaviour of ECCs under stress comes from the ability of the fibres to bridge the cracks. More precisely, the performance of ECCs is associated with the load transfer from the fibres to the matrix for the formation of successive cracks,
especially in the early stages of the applied load and later to the ability of the fibres to bridge the cracks holding the composite material together. However, for the composite to present a pseudo-ductile behaviour under tensile stress, the fibre and matrix need to be tailored in order to control the interface. The possible existence and strength of the bonding between fibre and matrix controls the failure mechanism, with strong bonding leading to fibre rupture and weak bonding to fibre pull-out: the key is to stop the fibre from pulling out too easily whilst minimising damage to the fibre.

Figure 2-10: Link between composite material constituents: fibre, matrix and interface to the composite tensile pseudo-ductile property (Li, 2002b)

2.4.6.3 Steady state crack analysis

The steady-state crack concept was first developed by Marshall et al. (1985), Marshall and Cox (1988) and then adopted by Li and Leung (1992) for ECCs. The steady-state crack analysis concept is associated with the analysis of the crack width, and suggests that a constant crack width over a wide range of tensile strain could be considered a material property and not a structural criterion performance.

Two different types of cracks can be described (Figure 2-11):

1) Griffith type crack: the crack increases in width resulting in the formation of a large crack after which the material usually fails. This can happen in the material if the fibre-cement interface is either too weak, so the pull-out of the fibres results in a low strength value of the
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composite material or the fibre-cement interface is too strong, hence, the pull-out of the fibres is associated with a low value of crack opening and results in a sudden failure.

2). Steady state flat crack: the crack remains flat, the fibres bridge the crack and transfer the load to the matrix leading to the formation of multiple (fine) cracks, i.e. a pseudo-ductile material. This is possible when the fibre-cement interface is neither too strong nor too weak. One of the most important conditions for the transition from brittle to pseudo-ductile behaviour is therefore the presence of steady state flat cracking.

The steady-state crack analysis also demonstrates the importance of the fibre-cement interface in the formation of the multiple fine cracks leading to the pseudo-ductile behaviour of the ECC material under stress.

Figure 2-11: Steady-state crack analysis revealing two different crack propagation scenarios with fibres (a) Griffith type crack and (b) Steady state flat crack (Li, 2002b)
2.4.6.4 Micromechanical parameters: fibre, matrix and interface

Whilst this section has not considered the practicalities of making a prediction about the behaviour of a particular mix and hence the process of optimisation, it can be seen that in order to obtain the desired pseudo-ductile behaviour under stress, the fibre, matrix and fibre-matrix interface have to be considered carefully. The details of the parameters controlling the pseudo-ductility of the composite under stress will be described in the next section, although at this point it is worth noting the following outcomes of Li’s (2002b) analysis:

- As the PVA fibres are relatively expensive compared with cement, their use in a low quantity, whilst maintaining a pseudo-ductile behaviour under stress, is preferred.
- A high fibre content can reduce the workability/consistency of the mixture (its ability to deform in its fresh state).

Therefore, the fibre volume fraction has to be chosen according to the fibres, matrix and interface properties and additional considerations such as cost and workability. Below a specific fibre volume fraction, pseudo-ductility of the material under stress cannot be observed resulting in a conventional FRC behaviour and adding fibres above this value increases the cost and decreases the workability of the mixture making the material processing difficult.

2.5 Mechanical Behaviour of ECCs

2.5.1 Introduction

Having introduced the key composite mechanics in relation with the behaviour of reinforced composites, an understanding of the specific mechanical behaviour of ECC is key with respect to design considerations, application (casting or pumping) on site and to some extent the longevity of the material. In section 2.3.4, pseudo-ductility was introduced as a phenomenon occurring in ECCs. In the current section, this phenomenon will be defined and discussed further in relation to the principles of micromechanics detailed in section 2.4; in particular the mechanism of pseudo-ductility will be outlined, together with the parameters which control this. Also of interest are the physical properties (i.e. density, porosity, fibre dispersion and orientation) as these will have an impact on the mechanical behaviour of ECCs.
2.5.2 Mechanism of pseudo-ductility

Figure 2-12 presenting the tensile stress and crack widths as a function of the tensile strain of ECCs, reveals that cast and flat specimens of ECCs (with a thickness of 12.7 mm) can, routinely, exhibit significant pseudo-ductility under tensile stress as a result of multiple-cracking of the matrix with a tensile strain of 5.3 % (Li, 2003). Tensile strains up to 8 % have been observed with the incorporation of polymeric fibres, which promote the formation of multiple and very fine cracks instead of large crack(s) leading to the premature failure of the specimen (Li, 2003). As illustrated in Figure 2-12, in comparison with a typical concrete, instead of a catastrophic failure at extremely low strains, the ECC material is able to exhibit pseudo-ductility under tensile stress, which is associated with multiple-cracking of the matrix, where much larger strains to failure are observed. This behaviour is due to the ability of the material to crack in multiple locations under tensile stress such that the strain is distributed across large numbers of cracks, which typically remain sub-100 μm in width prior to final failure (Li, 2003). Hence ECC exhibits high strain, which is often attributed to the increased number of cracks (rather than crack widths, being kept low). The higher the strain, the greater the number of cracks (at constant crack width) and the ultimate strength achieved is higher than the strength value at first crack. The desired behaviour of this material under stress is therefore a high number of fine cracks with a crack width not exceeding 100 μm. This means the tensile deformation is uniformly distributed in the specimen because the ECCs are pseudo-ductile under tensile stress. Figure 2-12 also shows the importance of the ACK Zone II (multiple-cracking zone) in the performance of ECCs compared to the ACK model detailed in section 2.4.

Hence, the pseudo-ductility of ECCs is enabled by the ability of the fibres to successively transfer the stress to the matrix via the fibre-matrix interface resulting in the formation of multiple cracks, limiting crack growth – and producing a large number of fine cracks as opposed to a single crack (Li, 1997). The length of fibres used will vary depending on the length scale of the other constituents in the mixture. In order to achieve such performances, the microstructure of the cementitious composite should be optimised using micromechanical models that take into consideration the mechanical interactions between the fibre, matrix and interface. The micromechanical models suggest the use of fibres with a diameter lower than 50 μm (Li, 2002a).
Most often, the pseudo-ductility of ECC is confirmed through a uni-axial tensile test. Previous studies on the properties of ECCs have tended to focus on thin specimens. For example, the Japanese Society of Civil Engineers (JSCE, 2008) reporting on High Performance Fibre Reinforced Cement Composites (ECCs being a typical example of this type of material), specifies the use of 13 mm thick specimens for testing ECCs in tension. In practice, ECCs might be used in structures of thicknesses of the order 50-150 mm. Hence, there is a need to move from initial work focussing on comparatively thin sections to thicker sections. Whilst thicker sections have been tested (Kanakubo, 2006; Kim et al., 2007), further work is still required to understand the effects of thickness and fibre orientation on strength.

In practice, structures manufactured from ECC are more likely to be loaded in bending than in pure tension, but given that samples in bending are still subject to stresses with a tensile component, multiple-cracking associated with the pseudo-ductile behaviour of the material is still expected. Topolar et al. (2012) present a model for a concrete material tested in four-point bending with three stages identified (Figure 2-13). There is an initial elastic stage up to the onset of cracking, which is followed by a period of (stable) micro-cracking up to the peak load, then the formation of a macro-crack which is (unstable) and causes stress relief. Where the material is unreinforced, the specimen will completely fail but when the concrete material contains fibres, the specimen re-stabilises and a fibre bridging process maintains the
component at this higher stress until complete failure.

Flexural strength (also called modulus of rupture) is one of the measures of tensile strength of concrete materials (NRMCA, 2000) and is an important parameter for designers. Flexural strength can be used as a measure of a cementitious material beam resisting failure in bending (NRMCA, 2000). A typical concrete shows a flexural strength of about 4.4 MPa, which increases to 6.4 MPa for a 50 mm ECC layered beam thanks to the presence of the fibres (Zhang et al., 2006). However, in order to evaluate the mechanical performance of ECCs in flexure, it is necessary to calculate the bending moment and curvature (JCI-S-003-2007). In such a test, the bending moment results in part of the beam being submitted to tensile stresses, hence the calculation of the curvature value being the difference of the strain in compression and tension, is a good indication of the ductility of the ECC material. The Japanese Concrete Society for testing HPFRCCs shows a typical bending moment-curvature curve compared with the tensile stress-tensile strain curve (Figure 2-14).

Figure 2-13: Fracture model for cement, plain concrete and fibre concrete tested in four-point bending (Topolar et al., 2012)
2.5.3 Parameters controlling pseudo-ductility

2.5.3.1 Introduction

Pseudo-ductility is the defining property of ECCs (section 2.3.4) and the one which, above all others, is key to its potential for tunnelling applications. One of the most useful properties of ECCs is their ability to undergo multiple cracking, a process in which the polymer fibres (at about 2\% by volume) are able to bridge the crack and carry the load without failure. The cracks that form open minimally and instead of failure, the component continues to carry the load with the formation of multiple-cracks. Indeed, pseudo-ductility is defined as the ability of the material to undergo a process of multiple cracking, such that the matrix of a sample or structure made from the material undergoes a process of successive cracking throughout the whole volume as increasing load is applied. Pseudo-ductility is dependent on the presence of short reinforcing fibres in the case of ECC. Hence, the mechanism is a function of fibre length, diameter and fibre surface coating, in addition to the fibre’s mechanical properties (Li et al., 2001). In turn, this means that there are a number of opportunities for influencing the mechanical behaviour of the ECC:

- Choosing a fibre with a particular set of properties;
- Controlling the fibre volume fraction;
- Controlling the fibre dispersion, distribution and orientation;
- Tailoring the fibre-matrix interface through the choice of fibre coating;
- Controlling the size of fibres and aggregates;
- Controlling the composite density and porosity and;
- Addition of controlled defects

In this section, the parameters influencing the pseudo-ductility of ECCs are detailed.
2.5.3.2 Fibre type and volume fraction

As noted, one of the applications for ECCs is as a secondary lining for hydraulic tunnels. The intention is that this material will not suffer from corrosion in service: by removing one significant mechanism for deterioration, structures will have an extended design life. This immediately reduces the choice of fibres available to use, but there is still a choice to be made. Given the commercial considerations of this project, the fibre of choice needs to be relatively inexpensive, whilst still delivering the required properties and being available in the required quantities over the course of time. ECCs have tended to focus on PVA fibres (Li, 2002a).

In terms of the volume fraction, the quantity of fibre included in the mix has to be sufficient to allow pseudo-ductility to occur, but not so great that the workability or consistency of the mixture is compromised. Research on ECC focusing on PVA fibres to improve the matrix properties, has shown that 2 vol. % is the ideal loading: below 1.5 vol. % and multiple cracking cannot occur, whilst a higher percentage of fibres (above 2.5 vol. %) reduces the workability and increases the cost (Li, 2002b). Li (2002b) has used the micromechanics\(^\text{10}\) approach to demonstrate how to obtain the desired pseudo-ductile behaviour while minimising the critical fibre volume fraction (sections 2.4.3 and 2.4.6). On this basis, most subsequent research has been carried out using a \( V_f \) of 0.02 (i.e. 2 vol. %), but in fact, a range of acceptability is likely to be \( 0.015 < V_f < 0.025 \).

2.5.3.3 Fibre aspect ratio

The fibre aspect ratio refers to the ratio \( L_f/d_f \). As detailed in section 2.4.3, both Naaman (1987) and Li (1997) demonstrated the influence of \( L_f \) and \( d_f \) in the determination of \( V_{f,crit} \), which are important parameters influencing the initiation of the pseudo-ductile behaviour of ECCs under stress.

Takashima et al. (2003) studying the influence of the fibre aspect ratio found that the greater the \( L_f/d_f \) ratio, the lower the fibre pull-out length value, resulting in higher ultimate tensile stress values for specimens. However, the optimal tensile strain is exhibited with mixes having an intermediate fibre aspect ratio. This study also shows that the pseudo-ductile behaviour of

\(^{10}\) Essentially an energetics approach to crack growth, showing how a finite volume fraction of fibres can limit crack opening and control failure through a bridging mechanism. From this, it can be added that a good fibre dispersion (and in some contexts, alignment) is essential to achieve pseudo-ductility.
ECCs, particularly the ultimate tensile stress is governed by the fibre pulling-out rather than fibre fracture. The theoretical models were detailed in section 2.4.3.

2.5.3.4 Fibre dispersion, distribution and orientation

Since the ability of ECCs to exhibit pseudo-ductility under stress is due to the presence of specially-designed micro-fibres enabling the transfer of load back to the matrix for the formation of successive cracks, the physical characteristics governing the presence of the fibres in the cementitious matrix such as fibre dispersion, distribution and orientation are essential.

Fibre dispersion relates to the good separation of the fibres in the cementitious matrix, i.e. it relates to the de-agglomeration of the fibres. The process of multiple-cracking is dependent on the ability to disperse these agglomerates during the mixing stage. Fibre distribution on the other hand relates to the final microstructure of the material. In order to achieve repeatability of the material, a quasi-homogeneous distribution is required in addition to the presence of fibres in all areas of the cementitious matrix. Some processing routes can lead to various kinds of heterogeneity being introduced, i.e. localised alignment, localised clustering or fibre poor areas. Hence, both characteristics play an important role in obtaining excellent pseudo-ductile behaviour of ECCs under stress. Although fibre dispersion and distribution are often confused with each other. Lee et al. (2009) state that the fibre dispersion within the cementitious matrix will play an important role in the mechanical performance of ECCs. Of all the factors affecting pseudo-ductility, fibre dispersion is the one that is in the hands of those mixing the mortar; in that sense, it is the factor that is most critical to the success of the material in practice, as well as to the on-going, batch-to-batch, quality control.

Lee et al. (2009) identifying the fibre dispersion as a crucial factor to achieve the maximum potential of ECCs, also suggested a way of evaluating the fibre dispersion using a fluorescence\textsuperscript{11} technique on a PVA fibre ECC, where the impact of the fibre dispersion could be correlated with the mechanical test results. In particular, Lee et al. (2009) determined a specific protocol to evaluate the fibre dispersion, where the images of the fibres are classified and categorised: different PVA fibre morphologies are illustrated in Table 2-4. An ideal fibre

\textsuperscript{11} A fluorescence image is taken with a charged couple device camera through a microscope and the fibre dispersion is analysed with image processing tools.
dispersion is distinguished by an important number of single fibres (S1), whereas fibre type N3 characterises a low fibre dispersion (which is characterised by fibre agglomeration). In addition to an ideal case where all the fibres are well dispersed (which also means that the fibre bundles are broken up into individual fibres), they should be equally distributed across the specific area analysed (or volume produced).

Table 2-4: Type of fibre images (Lee et al., 2009)

<table>
<thead>
<tr>
<th>Type</th>
<th>Number of fibers (thresholding algorithm)</th>
<th>Number of segmented objects (watershed algorithm)</th>
<th>Most probable number of fibres*</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single fiber</td>
<td>Type S1</td>
<td>1</td>
<td>1</td>
<td><img src="image1.png" alt="image" /></td>
</tr>
<tr>
<td>Possible single fiber</td>
<td>Type N1</td>
<td>More than 2</td>
<td>1</td>
<td><img src="image2.png" alt="image" /></td>
</tr>
<tr>
<td></td>
<td>Type N2</td>
<td>More than 2</td>
<td>1</td>
<td><img src="image3.png" alt="image" /></td>
</tr>
<tr>
<td></td>
<td>Type N3</td>
<td>More than 2</td>
<td>More than 2</td>
<td><img src="image4.png" alt="image" /></td>
</tr>
<tr>
<td></td>
<td>Type N4</td>
<td>More than 2</td>
<td>More than 2</td>
<td><img src="image5.png" alt="image" /></td>
</tr>
</tbody>
</table>

* These numbers are obtained from fluorescence image observations.

Fibre orientation on the other hand is characterised by its distribution in a three-dimensional space, more particularly in a fibre reinforced cementitious matrix, the direction of the fibre axis (compared with the loading(s) direction). Takashima et al. (2003) studying the mechanical properties of propylene discontinuous fibre reinforced cementitious composites confirmed that fibre orientation is one of the most influencing factors on the mechanical performance of fibre reinforced composites. This is also confirmed in section 2.4.4 which details the strength model for composite materials. Further, it is also important to consider fibre orientation, which can affect the measured properties and hence the manufacturing routes that lead to preferential alignment of fibres, which need to be understood. Alignment would be beneficial if the fibres need to withstand a single or dominant principal stress. However, where a multi-axial stress state exists, fibres oriented in more than one direction will be desirable.

As the fibre dispersion, distribution and orientation depend mainly on the manufacturing process, the mechanical performance should be evaluated along with these parameters, and then be linked to the manufacturing process.
2.5.3.5 Fibre-Matrix Interface

The pseudo-ductile behaviour of the ECCs is enabled by the fibre successively transferring the stress to the matrix via the fibre-cement interface resulting in the formation of multiple-cracks, hence the fibre-cement interface is essential to the mechanical performance of the ECCs under stress.

A study (Horikoshi et al., 2005) reveals the fibre-cement interface as a layer of Ca(OH)$_2$ (from Ca$^{2+}$ and HO$^-$ ions present in the cement slurry) shown as a white colour and called the ITZ (Interfacial transition zone) surrounding PVA fibres as observed in Figure 2-15. This does not occur in the case of PP fibres, suggesting that the PVA fibre interacts with the matrix, leading to the formation of an interphase. Horikoshi et al. (2005) present a model showing the PVA interacting with the matrix to form a complex cluster with metal hydroxides (Figure 2-16). The presence of the interphase (and by inference, the strong interaction between the PVA fibre and cementitious matrix) is not disputed, but the model for the interaction is extremely basic and there is a lack of supporting evidence for the interaction to develop in this manner.

![Image of interface between PVA fibres and cement shown as a white colour (Horikoshi et al., 2005)](image-url)
Another study (Rathod and Patodi, 2010) suggests that hydrophilic PVA fibres form a very strong chemical bond with cement due to the presence of hydroxyl groups on the carbon backbone in its molecular chains. The hydroxyl group leads to a strong hydrogen intermolecular bond, due to chemical adhesion, which is not beneficial for ECCs as the fibres will tend to rupture under tensile stress instead of pulling-out. On the other hand, a hydrophobic fibre, such as polypropylene, does not bond chemically to the matrix, hence frictional bonds govern the pull-out mechanisms. Rathodi and Patodi (2010) suggest the use of another type of fibre with a need to use properties between hydrophilic and hydrophobic in order to control the interface properties.

The mechanical performance of the ECCs is often characterised by the ability of the fibre to pull-out within the cementitious matrix under tensile load instead of breaking. For this reason, the surface of the fibres should be treated in order to reduce the fibre-cement bonding and to enable the pulling-out of the fibres from the cementitious matrix under stress. Hence, according to the literature (Li et al., 2002; Li, 2002b), the PVA fibres used in the study have been specifically tailored to pull-out rather than break when ECC samples are loaded in tension (Li, 2003). Tailoring the fibre and interface, also means ensuring a good choice of fibre type and properties, but also the fibre surface characteristics so as to maintain the desired interface associated with the mechanical performance for the specific application of the ECCs. A study conducted on PVA fibres supplied by Kuraray (Li et al., 2002) demonstrates the link between the frictional bond, the fibre surface coating (an oiling agent) content and the tensile strain capacity of the cementitious composite material, in order to obtain the desired ultimate
strain (Figure 2-17). Micromechanics estimated an optimal range of frictional bond $\tau_0$ to be between 1 and 2 MPa for the composite (ECC containing 2 vol. % PVA KII) to achieve pseudo-ductility. A single fibre pull-out test showed that this could be achieved with a surface coating content between 0.6 and 1.2 % as a target (Figure 2-17a): the ultimate strain for a sample tested in uniaxial tension increases with the coating content, where the highest strain is achieved with 1.2 % of coating content (Figure 2-17b).

Figure 2-18 compares the performance of composites made with fibres (a) without any coating content and (b) with fibres having a surface coating content of 1.2 vol. %. The low pseudo-ductility of the composite made with uncoated fibres is, according to the study, due to fibre rupture under stress. In all-likelihood, this is due to the existence of a strong chemical bond between the fibre and cement matrix: the presence of a coating enhances the ductility of the composite, reduces the strength of the bond, allowing the fibres to slip. The critical factor with such a coating therefore is to ensure that sufficient bond strength remains to hold the material together, but that it is not so great that it causes the fibres to fail without slipping.

Figure 2-17: Micromechanics determine an optimal range of bond $\tau_0$ of 1-2 MPa for the PVA KII fibre to achieve pseudo-ductility with 2 vol. % fibre content. (a) Single fibre pull-out testing shows that this target range can be achieved with a surface coating (an oiling agent) content between 0.6 and 1.2 %, (b) Composite uniaxial tensile test confirms increase of tensile strain capacity from 1 to 5 % when the surface coating content reaches 1.2 % (Li, 2002b)
Therefore, understanding - and controlling - the fibre-cement interfacial bond is valuable in promoting the pseudo-ductility of the composite material under tensile stress. Redon et al. (2001) examined the appearance of fibres at different stages of pull-out from a cementitious matrix (the aspect of the fibre pulled is linked with its mechanical performance); at large strains and close to failure, the ends of the pulled-out fibres exhibit thinned “valleys” of missing PVA on its surface and the embedded end is narrower than the original.

In this study, it will also be verified whether the fibres suggested by Li and which have been tailored for ECC are indeed able to lend the composite material a pseudo-ductility under stress and whether the frictional bond values suggested and measured by Li are indeed adequate when compared with the values measured through single-fibre pull-out tests from a cementitious matrix as conducted in this study. The test methods and results are detailed in Chapters 3 and 4, respectively.

Redon et al. (2001) suggest an excellent method to estimate the interfacial properties of PVA fibres in an ECC matrix. These are identified with single fibre pull-out testing from a cementitious matrix. According to this study, a typical fibre pull-out test curve enabling the identification of the bond properties of PVA fibres in a mortar matrix, can be characterised by three stages (Figure 2-19):

- Initial stage (Figure 2-19a): the debonding process commences along the fibre/matrix interface where the load increases to $P_a$ and the embedded end of the fibre does not move. The displacement recorded in Figure 2-19 corresponds to the elastic stretching
of the debonded fibre segment.

- Intermediary stage (Figure 2-19b): the load decreases from $P_a$ to $P_b$, a sudden decrease of the load shows that the chemical bond between the matrix and the fibre is broken (particularly in the case of PVA fibres). This could mean that a fracture criteria rather than a strength criterion governs debonding at the interface (Leung and Li, 1990; Li and Stang, 1997)

- Final stage (Figure 2-19c): the slippage regime where the fibre load is resisted by frictional forces (Redon et al., 2001). Once the fibre is fully debonded, this stage corresponds to the slippage of the fibre from the matrix in which the displacement increases up to the fibre embedded length $l_e = 0$; this stage could be characterised by a slippage coefficient $\beta$. Three cases can exist: a slip-hardening ($\beta > 0$), constant friction slippage ($\beta = 0$) or a slip-softening ($\beta < 0$).

On this basis it is possible to determine $G_d$, the chemical debonding energy (equation 2.45), and $\tau_0$, frictional bond strength (equation 2.46) from a single fibre pull-out test curve (Figure 2-19) as follows:

\[ G_d = \frac{2(P_a - P_b)^2}{\pi^2 E_f d_f^2} \]  
\[ \tau_0 = \frac{P_b}{\pi d_f l_e} \]
where $E_f$ is the Young’s modulus of the fibre, $d_f$ is the diameter of the fibre, $l_e$ the fibre embedded length and $P_a$ and $P_b$ are the single fibre pull-out loads at the points A and B respectively as indicated in Figure 2-19.

Redon et al. (2001) suggest that, because polymeric fibres are less hard than the surrounding matrix, one is more likely to observe slip-hardening behaviour (Figure 2-20a); however the fibre can also rupture before the complete pull-out from the matrix (Figure 2-20b) in the slippage regime.

Another characteristic that is of importance in characterising the fibre-cement bond is $\tau_d$, the interfacial shear strength:

$$\tau_d = \frac{P_a}{\pi d_f l_e}$$  \hspace{1cm} (2.47)

Zhandarov and Mäder (2005) use this property to assess the quality of the interfacial bond between a fibre and a cement matrix.

Finally, although applied to a specific type of fibre, the theoretical tools developed could be applicable to a wide range of fibre types and brittle matrix types. One of the aims of the present study will be to identify the current fibre-cement interfacial properties and to correlate this with previous studies. The identification of these would be correlated with the
mechanical performance of the ECCs. Also, it is intended to identify the chemistry of the interface/interphase between fibre and cement.

2.5.3.6 Fibres and Aggregates

Figure 2-21 shows the physical correlation of the fibres and the fine aggregates for crack bridging behaviour to occur, demonstrating the importance of their relative sizes, which would enable the bridging of the crack as it opens. The microscopic tailoring could also enhance the composite ductility.

Furthermore, Figure 2-21 also exhibits different bridging zones where the different constituents “work together” to bridge the crack forming. The whole interaction occurs across the “Fibre cement cohesive zone”, thanks to which the increase in crack width is prevented. It should be noted that for ECCs, sand is used instead of coarse aggregates, which has a size in the micrometre range, therefore the fibre characteristics have to be considered with respect to the size of the fine aggregates, which themselves have little or no contribution to the fibre bridging mechanisms. This needs to be taken into account to enhance the ECC performance.
2.5.3.7 Microstructure, Density and Porosity

Although there are not many publications linking the physical properties with the mechanical performance of ECCs, it is expected that the microstructure of the material would affect its mechanical performance.

According to Felekoglu et al. (2009), the size of the pre-existing flaws and defects in ECC would determine the critical load required for crack initiation. As expected, the existence of flaws would make the matrix more brittle and would promote matrix cracking at lower loads. It is also believed that a first matrix crack at lower loads would promote a higher toughness, however subsequent multiple cracking occurs thanks to the load transfer from the fibre to the matrix. Hence, the initial flaw and fibre dispersion would both play an important role on the pseudo-ductility (toughness) of the cement composite under stress. This observation also shows the importance of the microstructure of the material controlled by the composition and manufacturing process.

For example, porosity could expedite cracking, promoting “apparent” premature brittle failure under stress. In concrete technology, it is known that there is a link between the material’s physical properties and its mechanical behaviour under load. Indeed, a high density, associated with low porosity, usually indicates greater strength than low density and high porosity. Nail (1997) takes this further, showing that the durability of the material can also be strongly influenced by its density and/or porosity. In particular, great permeability of the material to aggressive agents is promoted by the presence of a high porosity which is usually inversely proportional to the concrete density.

2.5.3.8 Addition of Controlled Defects

Counter-intuitively, it is actually helpful to introduce defects (to act as stress concentrators) in order to limit the tensile failure strength of the cement matrix so as to achieve the maximum crack density and hence increase the strain capacity. The normal defect population would of course consist of pores (naturally occurring within ECC material) whilst an engineered defect population could be introduced in addition to this (Wang and Li, 2004). However, this has potential consequences for the permeability of structures manufactured from ECCs, even if the structure is un-cracked. This is because the engineered defect population may act to transport fluids through the section, particularly when such defects represent quasi-
connected porosity. In a water carrying structure, such behaviour could represent a significant limitation. In the context of the work presented in this section, the focus is on applying ECCs in an industrial application, where the achievable strain is a function of the crack density and the crack width. Wang and Li (2004) expressed the crack saturation in ECCs as a function of the existing flaws within the material.

2.6 Durability

2.6.1 Introduction

For the intended application of ECCs, a design life of 150 years should be considered which is specified by potential customers as a project-specific requirement. Eurocode BS EN 1990:2002 mentions a design life of 120 years as a minimum for permanent works, for which the material should continue to exhibit pseudo-ductility under stress. In concrete technology, the term durability often refers to the material being able to withstand the deterioration process to which it is exposed due to external factors or internal causes within the material itself (Neville, 2000). However, for ECCs, durability also refers to the maintaining of the pseudo-ductile behaviour of the material under stress associated with the ability of the fibres to pull-out in the cementitious matrix under load. Therefore, the long-term durability of the composite material is related to the ageing process of the cement and fibres, particularly to the stability of the fibre-cement interface (Van Zijl, 2005; Van Zijl et al., 2012). Finally, the durability of the ECCs can be evaluated as the ability of the material to heal itself once initial cracks appear, without further intervention post-casting. In this section, the long-term durability of the material is first presented as the material being able to withstand the deterioration processes to which it is exposed (longevity), then the maintaining of the pseudo-ductile behaviour with time linked to the material itself and the fibre-cement interface and finally, the ability of the material to self-heal once cracks appear.

2.6.2 Longevity

ECCs may be used in structures with design lifetimes of 150 years. As a simple definition, the durability of the ECC is the ability to maintain its structural strength and ductility for the intended period. It should be able to resist the deterioration processes to which it will be exposed when in service, which can be due to internal or external causes. Therefore, in order

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12 Strength associated with the on-going integrity of the structure, i.e. the arrangement of the parts or elements forming something complex such as tunnels, buildings
to evaluate the durability of this new type of material, it is essential to understand the causes of deterioration of ECCs: it should be noted that some mechanisms of deterioration are specific to a particular composition. Neville (2000) states that there are three main deteriorating factors for concrete structures when in service: mechanical, chemical and physical types (Table 2-5).

<table>
<thead>
<tr>
<th>Type of damage</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanical</td>
<td>impact, abrasion, erosion or cavitation</td>
</tr>
<tr>
<td>Chemical</td>
<td>alkali-silica and alkali-carbonate reactions (primarily on aggregates), external attacks: action of aggressive ions such as chloride, sulphates or CO$_2$, many natural or industrial liquids and gases</td>
</tr>
<tr>
<td>Physical</td>
<td>high temperature, difference in thermal expansion of aggregates and of the hardened cement paste</td>
</tr>
</tbody>
</table>

In the light of these factors, many authors such as Van Zijl et al. (2012) associated the durability of the ECCs (i.e. the absence of deterioration/failure) with the appearance of fine cracks instead of larger cracks, enabling the ECCs to resist the migration of aggressive substances and maintain the integrity of structures. According to Eurocode 2 (BS EN 1992-3: 2006), for structures designed to convey water, crack widths should be maintained between 0.05 mm to 0.2 mm depending on the hydraulic gradient for the water to be retained by the containing structure. If the crack widths can be maintained between these values, then it is assumed that the cracks would seal themselves (self-heal) in a very short time and the material could be considered as durable (i.e. maintaining structural integrity with time). Although ECCs are designed to exhibit crack widths below 0.1 mm, the use of ECC has to be considered carefully, as the durability of the material is also associated with the aging process at the fibre/cement interface.

A piece of work on ECCs (Jesson et al., 2009) suggested a possible chemical attack within the cementitious material. For example, in order to avoid any possibility of alkali-silica reaction leading to long-term expansion and cracking, the aggregates should be non-reactive and a low alkalinity cement should be used. Additional properties in relation with the possible degradation of the material, such as permeability and porosity of both cracked and un-cracked
material, also need to be investigated.

Generally, the durability of cementitious materials is improved when the ratio of water to cement is below 0.45, as the material is less permeable, which could also lead to high compressive strengths (Neville, 2000). In addition, compared with concrete material where a high compressive strength is required as it is associated with structural integrity and durability, the ECCs should exhibit excellent behaviour in tension over time; i.e. the maintaining of a high toughness.

An interesting study carried out on the degradation of PVA fibres revealed promising results (Lhoneux et al., 2002). PVA fibres in saturated cement solutions appear to show a decrease in tensile strength only at temperatures above 40 °C. In addition, the tensile strength of fibres extracted from aged samples taken from sites in Switzerland and Belgium, showed only a minor reduction in strength (1100 MPa after 18 years, compared with a value of 1200 MPa for as-received fibres). The study also revealed a good embedment of the fibres in the cementitious matrix, with only a small amount of micro-porosity. Although there is some evidence of the durability of PVA fibre-cement products, further investigation is required in the mechanisms of degradation. Moreover, although some accelerated aging tests such as “Beschleunigt Alterung CO2” or “BAC” using high temperatures (Lhoneux et al., 2002) were conducted, a specific durability testing method for ECCs is not established and further investigation is required.

### 2.6.3 Pseudo-ductility

An important aspect of the long-term durability of ECCs is the maintaining of the pseudo-ductile behaviour over time. The relevant conditions for the composite to exhibit a pseudo-ductile behaviour under stress are stated in section 2.4.3 and reproduced below:

- Naaman and Reinhardt (1996):

\[
\sigma_{fu}V_f \geq \sigma_{mu}(1 - V_f) + \varepsilon_{mu}E_fV_f
\]  

(2.48)

Equation 2.48 implies that the fibre is able to support the load once the matrix cracks. Therefore, in order for ECCs to exhibit a pseudo-ductile behaviour with time, the fibre strength should be greater than the matrix at 150 years; the condition for pseudo-ductility stated in
equation 2.48 must be valid with time. As the strength of the cementitious matrix and the associated Young’s modulus increase with time following a specific pattern (Rossi, 2013), it is possible to determine the tensile strength of the cement at 150 years and verify the condition above.

- Naaman (1987) states that the post-cracking strength $\sigma_{pc}$ should be higher than the strength at first cracking $\sigma_{cc}$

$$\sigma_{pc} \geq \sigma_{cc} \quad (2.49)$$

where:

$$\sigma_{cc} = \sigma_{mu} (1 - V_f) + \alpha \tau_0 V_f \frac{L_f}{d_f} \quad (2.50)$$

$$\sigma_{pc} = \lambda \tau_0 \frac{L_f}{d_f} V_f \quad (2.51)$$

Verifying the two conditions presented in equations 2.48 and 2.49 will give an idea of whether the pseudo-ductility is maintained with time, based on the assumption that the fibre characteristics such as strength and physical aspect remain constant with time. The prediction of pseudo-ductility over time is, however, somewhat complicated by the evolution of the fibre-matrix interface over time and this must be borne in mind when designing on the basis of such a prediction.

### 2.6.4 Fibre-cement interaction/interface

An important issue associated with the long-term durability is the ageing processes at the fibre-cement interface, which can have an impact on both stress and strain at first crack and further cracking. The ageing processes at the interface is controlled by the chemistry of the fibre-cement interface, which could influence the pseudo-ductility of the material over-time, and therefore the durability of ECCs.

The discussion in section 2.5.3.5 also applies, to some extent here. The strong chemical bond between the PVA fibres and the cementitious matrix discussed by Li et al. (2004) will change
over time, becoming stronger. If the bond strength increases with time, then under tensile stress, the fibre within the ECC will more likely break instead of pulling-out, which could reduce the pseudo-ductility of the ECC in the long-term, potentially compromising the long-term durability. Horikoshi et al. (2005) state that the PVA fibre reacts with the cementitious matrix to form an interfacial transition zone (an interphase, primarily composed of Ca(OH)$_2$) between the fibre and the matrix.

In order to reduce the bonding between the fibre and cement, the fibres used in this study have been specifically tailored with a coating so as to pull-out from the cementitious matrix rather than break when ECC samples are loaded in tension (Li, 2003), as discussed in section 2.5.3.5. Hence, the coating placed on the surface of the fibres needs to be maintained over time in order for the desired behaviour of ECC under stress to be preserved. This will be investigated and discussed in Chapter 5 in relation to the durability of the material.

Single fibre pull-out tests at different times would give interesting information on the interfacial interaction between fibre and matrix (Wood, Smith and Watts, 2007). For instance, characterising the changes with time of the frictional bond strength ($\tau_0$), the interfacial shear strength ($\tau_d$) and the chemical debonding energy ($G_d$) would contribute to the understanding of the long-term durability of ECCs. Often fibres have been coated with an oiling agent so as to control fibre-matrix bonding and therefore, the long-term durability is dependent on the stability of the coating, which controls the interface over time.

Horikoshi et al. (2005) provided interesting data on the strength of a PVA fibre reinforced cementitious matrix material exposed to the external environment for nearly 18 years (Figure 2-22). It was found that the material flexural strength is maintained and/or is higher than before aging (Figure 2-22) and no sign of degradation was found on the fibres extracted from the fibre cement sheet exposed to outdoor weathering. However, it is not known whether the fibres had been treated in the past before being included in ECC. And in any case, whether the fibre-cement bond strength changes or not with time does not seem to have a negative influence on the mechanical properties of the material. The resistance to the flexural deformation (flexural strength) is maintained and/or increased; however there is no indication of the ultimate strain capacity of these samples.
A study (Horikoshi et al., 2005) on accelerated aging showed that PVA fibres soaked in hot (80 °C) and alkaline water maintained their tensile strength even at 14 days soaking (Figure 2-23). The authors also demonstrated that PVA fibre reinforced mortar specimens, soaked in 80 °C hot water, kept their modulus of rupture. However, there are some concerns on the validity of these aging tests. As explained earlier in this section, the durability of ECC is also closely linked with the fibre-cement interface and an increase in temperature is not a real indication of the possible change in the fibre-cement bond strength and interface chemistry with time, which is a complex phenomenon when it comes to ECC.
2.6.5 Self-Healing

The durability of ECCs can also be evaluated in terms of the ability of the material to heal after the appearance of initial cracks, whether these cracks are due to shrinkage upon curing or due to applied loads in service at any time during the design life. However, for this to happen, the width of these cracks must not exceed the threshold value required for healing, which is below 150 µm and preferably, below 50 µm (Yang et al., 2009). Larger cracks could permit water and gas ingress, and compromise the durability of the lining.

Healing can be achieved via a number of routes. Healing can come from the material itself: this phenomenon is called autogenous healing or self-healing. The ability of ECCs to self-heal is thought to be due to the presence of unhydrated cement that hydrates under wet conditions, filling the voids created by the cracks. It corresponds to the hydration of unreacted cement (Yang et al., 2009). Yang et al. (2009) presented the self-healing capacity of ECCs when submitted to wet and dry cycles after the appearance of initial cracks. ECCs are, by design, high cement content materials and there is usually a proportion of the cement particles that remain unhydrated. As the cracks formed are very fine, usually with a maximum width of 100

Figure 2-23: Strength retention of fibres versus the soaking days in cement at 80°C (Horikoshi et al., 2005) – Kuralon and ARG refer to Polyvinylalcohol (PVA) and Alkali Resistant Glass respectively.
µm, it is relatively easy for the cracks to be filled with products from the unhydrated cement available. Hence, the quantity of unhydrated cement is relatively important in the autogenous healing ability of the material, but it should be noted that it is a finite resource, which can become exhausted if the structure is subjected to recurring sub-critical damage.

Healing can also be achieved by adding new components to ECC:

- **Microcapsules** - these are small capsules that release gel or water when cracked, leading to hydration of unhydrated cement or crackfilling by a polymer healing agent (Wu et al., 2012; Figure 2-24).
- **Bacteria** - Wu et al. (2012) suggested the use of bacteria (considered as a pollution free and natural repair technique) in the cementitious material which would help the precipitation of calcium carbonate (Figure 2-25).

![Figure 2-24: Healing of cracked material by incorporation of microcapsules (a) Schematic illustrating (i) the crack approaching the microcapsule, (ii) the healing agent released when the microcapsule cracks and (iii) the healing agent reacts with catalyst to fill voids created by the crack, (b) SEM image of a ruptured microcapsule (Wu et al., 2012)](image-url)
Of these mechanisms, autogenous healing is likely to be the most practical factor, especially in the context of a hydraulic tunnel transporting potable water; the addition of another constituent is not feasible, particularly if it may lead to contamination of the contents of the tunnel.

However, Yang et al. (2009) suggested that the autogenous healing ability of ECCs subjected to wet-dry cycles could be due to one of a number (or a combination) of several different mechanisms (Figure 2-26), including hydration of the unreacted cement, expansion of concrete (swelling of CSH), closure of cracks by solid matter (such as impurities present in water) and by crystallisation of calcium carbonate (CaCO$_3$). For mature cementitious materials, the last is considered as the main mechanism for self-healing, probably because the material is more prone to the ingress of CO$_2$ within its structure.

Figure 2-25: Scenario of crack healing with bacteria: the material cracked is placed in water, the ingress of water in the crack activates the bacteria which then multiply and precipitate minerals such as CaCO$_3$ (Wu et al., 2012)
Figure 2-26: Possible mechanisms of self-healing in cementitious materials (a) formation of CaCO₃ or Ca(OH)₂, (b) presence of impurities or loose concrete particles blocking the crack, (c) hydration of unreacted cement, (d) expansion of hydrated cementitious matrix (swelling of CSH) (Wu et al., 2012)

Resonance frequency measurements (a non-destructive method to determine the level of damage that has occurred to a sample) have been used to quantify the self-healing ability of ECC samples subjected to wet-dry cycles (Yang et al., 2009). Cracked ECC samples have been shown to recover 76 to 100% of their initial resonant frequency, which, when taken with tensile test data indicated that specimens that have undergone an autogenous healing process can recover up to 100% of their undamaged strength. Based on initial Energy Dispersive X-ray spectroscopy (EDS) studies, the mechanisms of self-healing could be related to the growth of calcites (calcium carbonate crystals) in the cracks (Yang et al., 2009), suggesting that the hydration of un-reacted cement may not always be the main healing mechanism, but this is less likely for ECC containing a high content of un-reacted cement. Also the precipitation of calcium carbonate necessitates the presence of CO₂, which depends on the environment and quality of the material.

The ability to heal, and the longevity of this ability, is clearly dependent on the quantity of the constituent involved in the healing mechanism, whether it is polymer filled micro-capsules or some route leading to the hydration of unreacted cement. Therefore, to evaluate the ability of ECCs to autogenously heal, the presence and quantity of unhydrated cement needs to be examined. According to Diamond (2004), the microstructure of the cementitious material can be visualised clearly in scanning electron microscopy (SEM) using the backscattered electron mode. The backscatter mode is one of a number of different SEM imaging techniques. The technique is based on the atomic number (Z) contrast, which means that elements with a low Z will appear black while those with high Z will be white/lighter.
Compounds/structures with a mix of high and low Z atoms will lead to some shade of grey: in backscattered electron imaging, the available ‘grey scale’ ranging from white to black, is usually divided into 256 ‘grey levels’ or shades of darkness. An example of a backscattered electron image of a cement paste is illustrated in Figure 2-27, showing a very old specimen (100 years old) of cured cement paste still exhibiting unreacted cement, which has been prevented from reacting by the formation of hydration product shells. The presence of these particles could be even more significant for low water/cement ratio pastes such as those formed by ECC, although it could be argued that a 100 years old cement composition is slightly different to modern PC (EN197).

![Figure 2-27: View of the microstructure of a 100 year old cement paste (w/c = 0.30) cured at room temperature (Diamond, 2004)](image)

The unhydrated components in cement have much higher electron backscatter coefficients than the hydrated products and so the residual unhydrated cement grains appear in backscattered electron SEM images as bright entities in a sea of darker areas (Diamond, 2004). The un-hydrated cement particles can therefore easily be identified by this typical feature, where the clinker components are all crystalline with typical grain sizes ranging from about 1
µm to 60 µm. It is also straightforward to observe the remaining unhydrated material and to distinguish between particles that are completely hydrated and those that are only partially hydrated.

2.7 Shrinkage and Water-tightness

2.7.1 Introduction
From the early ages of its life, a cementitious material undergoes volume change, which manifests itself as shrinkage (Holt, 2001). Shrinkage exists as a result of the hydration of cementitious material: chemical and physical changes occurring when water is added to the dry material. The degree of shrinkage depends on the temperature, humidity, air flow and also the composition of the material. Of particular interest to both the practical application of this material as well as a way of controlling cracking, shrinkage of the ECCs is investigated. Shrinkage is particularly associated with initial curing of cast material, but may occur later in the life of the structure, typically because of a long-term change in use or environmental condition (e.g. a hydraulic tunnel taken out of use and drained for an extended period). Ideally, ECC materials should not present any shrinkage for their application which is not realistic, hence the design value for shrinkage of ECC materials is taken to be lower or equal to the shrinkage of concrete.

Different types of shrinkage need to be considered in the short and long-term (Holt, 2001). The longevity of a structure depends on the intensity of these types of shrinkage with time (Gilbert, 2001). However, for ECCs, drying shrinkage should be particularly investigated as it is the most important factor affecting cracking of the ECCs (Zhang et al., 2009), especially as the material is restrained when used as a secondary lining in tunnelling. In this section, different types of shrinkage (and their related issues such as cracking) will be examined and discussed followed by a discussion on the influencing parameters and finally details of the methods for controlling shrinkage such as the use of additives.

2.7.2 Plastic shrinkage
Plastic shrinkage characterises the shrinkage of the cementitious material when it is in the plastic state. It occurs in the early ages of the material and corresponds to the loss of moisture to the surrounding environment through evaporation before the material hardens. The magnitude of plastic shrinkage is therefore affected by the amount of water lost from the
surface of the cementitious material, which is dependent on temperature, ambient relative humidity and air flow (Neville, 2000).

Wongtanakitchaoren and Naaman (2007) recognised that in restrained conditions, the tensile stresses developing within the material due to shrinkage lead to potential cracks, obviously known as plastic shrinkage cracks. Free shrinkage of the material at early age could also lead to the development of tensile stresses within the material, although not necessarily resulting in cracking (Boshoff and Combrinck, 2013). Cracks due to plastic shrinkage under restrained conditions are a significant problem that occur in concrete structures (Wongtanakitchaoren and Naaman, 2007), producing a direct path for the ingress of any fluid into the structure. This reduces the performance, serviceability and durability of the structure making it vulnerable if further stresses due to loading are applied.

Plastic shrinkage cracks are mainly observed in thin elements with a high surface area to volume ratio and/or if the evaporation rate exceeds the bleeding rate, causing tensile stresses to develop in the capillary pores, which may be higher than the tensile strength of concrete particularly at early ages. Neville (2000) states that plastic shrinkage cracking occurs if the amount of water lost per unit area exceeds the amount of water brought to the surface by bleeding\(^{13}\), and is large.

Plastic settlement due to especially early initial water loss could also lead to the formation of cracks during the early age setting of the material (known as plastic settlement cracking). However, the mechanism is different. Due to gravity, the dense particles will tend to move downward whilst water migrates to the top of the material structure, resulting in some settlement. The cause of plastic settlement cracks may occur when settlement is constrained by a reinforcement rod for example (Chu, 2010). Plastic shrinkage cracking and plastic settlement cracking can sometimes be confused with one another (Neville, 2000). However, cracking due to plastic shrinkage can be reduced and even prevented by reducing rapid drying of the surface of concrete material as well as by the use of fibres as a secondary reinforcing mechanism (Sivakumar and Santhanam, 2007).

\(^{13}\) Bleeding is a form of segregation in which the water added to the mixture tends to rise to the surface of fresh material, due to the inability of the solid constituents to hold all of the mixing water (Neville, 2000). The bleeding rate is the rate at which the water moves through the plastic concrete.
For ECCs, where shrinkage cracking could be a problem, reducing plastic shrinkage by preventing the evaporation of water from its surface could be a solution, or alternatively finding a way of accelerating the hardening of the ECCs and limiting the duration of its plastic state could help. Due to the high cement content of ECCs and the addition of fibres, ECCs would set relatively quickly and therefore plastic shrinkage cracking may not be a major issue compared to the other types of shrinkage, probably because there is less water to evaporate. Bentz et al. (2009) confirmed the latter view, as they found that setting times of cement-based materials vary by several hours when four different water/cement ratios were tested, as the water/cement ratio controls the volume of water available for hydration per unit volume of cement.

Wongtanakitcharoen and Naaman (2007) studying the influence of fibres on water evaporation and free shrinkage of concrete during the first 24 hours after the placement of concrete found that the use of fibres such as PVA reduced the plastic shrinkage deformation by 36.20 %, and 0.1 % fibres by volume is sufficient to reduce unrestrained shrinkage strain, and this without any particular influence of the physical aspect of the fibres. Mangat and Azari (1984) explained that when shrinkage induces tensile stresses within the matrix, fibres are able to restrain shrinkage by shear along the fibre-matrix interface and by making the composite material stronger and stiffer.

2.7.3 Drying shrinkage

Drying shrinkage corresponds to the volume change due to the drying of cement-based materials, post-setting, as water evaporates to the atmosphere through diffusion. Neville (2000) defines drying shrinkage as the removal of water from concrete when stored in air but notes that the change of volume in drying concrete is not equal to the volume of water removed, which suggests that additional mechanisms are at work such as chemical shrinkage (associated with the chemical reactions between cement and water). The mechanism by which shrinkage happens is complex but is thought to involve a combination of water loss from the large capillary pores which does not cause significant volume change and then from the small capillary pores and subsequent loss from the gel pores (Cement Concrete and Aggregates Australia, 2002). A part of this shrinkage is irreversible and should be distinguished

\[14 \text{ Setting refers to the stiffening of the cement paste (Neville, 2000). Once the cement powder is mixed with water, the cement paste gradually stiffens and hardens.} \]
from the reversible moisture movement caused by alternating storage conditions between wet and dry. Thomas and Jennings (2005) specified that the driving force for drying shrinkage is capillary tension in the pores and disjoining pressure between the particles of cement which are reversible and that irreversible shrinkage results from physical and chemical changes to the paste, which are generally the rearrangement of the C-S-H gel structure, however the irreversible part of shrinkage is mostly affected by ageing. Of all forms of shrinkage, drying shrinkage in particular can have a significant impact on the physical properties such as permeability and porosity and durability as well as mechanical properties such as strength. Zhang et al. (2009), investigating a method of reducing shrinkage, noted that drying shrinkage is the most important factor affecting cracking of ECCs.

Drying shrinkage continues for the whole life of the cementitious material composite, although most of the drying shrinkage occurs during the first 90 days. It can be affected by the particular constituents used, such as the volume of the aggregates (smaller particles will have the tendency to increase shrinkage), or the water/cement ratio (an increase could promote shrinkage) and by environmental conditions such as temperature and humidity. Holt (2001) estimates that 80% of laboratory measured shrinkage occurs within about three months and the amount of drying shrinkage is controlled by both the environment and properties of concrete. The duration of shrinkage would also depend on the size and shape of the concrete element as well as the environment controlling the rate of moisture loss. Holt (2001) gives the following equation in accordance with the CEB-FIP (1990) model code, which predicts the magnitude of shrinkage of a concrete specimen at a specific time $t$ which depends on several parameters:

$$\varepsilon_S(t,t_0) = \varepsilon_{S0} \left[ \beta_S(t) - \beta_S(t_0) \right]$$

(2.52)

where $\varepsilon_{S0}$ is the basic shrinkage coefficient ($= \varepsilon_1 \times \varepsilon_2$), $\varepsilon_1$ is a factor depending on the environment, $\varepsilon_2$ is a factor depending on $h_0$, $h_0$ is the notional thickness depending on specimen size and ambient humidity, $\beta_S$ is a factor for shrinkage development with time, depending on $h_0$, $t$ is the concrete age if temperature is different from 20°C and $t_0$ is the age when drying starts. Hence, larger specimens will shrink for longer periods but the ultimate magnitude may be lower.
Equation 2.52, established for concrete, mainly depends on the environment, physical aspect of the specimen and a factor for shrinkage development with time depending on the notional thickness, temperature and age. Although concrete usually exhibits a lower drying shrinkage than ECCs, the equation could probably be adapted to ECCs.

Shrinkage is usually measured on a linear scale; the conversion from linear measurements to the volume change is made using the following equation (Holt, 2001):

\[
\text{Volume Change} = 1 - \left(1 - \frac{\text{shrinkage}}{\text{length}}\right)
\]

According to Holt (2001), drying shrinkage of concrete also depends on the porosity, especially the internal pore spaces. The microstructure includes solid particles, pore size and capillary voids, the latter being the spaces occupied by the water removed during the hydration mechanisms. Therefore, controlling the material microstructure would enable a degree of control over drying shrinkage.

It has been suggested that the addition of non-metallic fibres reduces the drying shrinkage crack widths at later ages (Sivakumar and Santhanam, 2007). Passuello et al. (2009) demonstrated that the addition of fibres increases the resistance to crack opening in the case of drying shrinkage, the study found that crack opening is reduced by 90 % with the use of micro-fibres and this by transferring the stresses across the cracks, hence limiting the crack width. The effectiveness of PVA fibres in controlling plastic/drying shrinkage cracking in ECCs has been demonstrated by Li et al. (2001). However, only a small amount of fine sand was used in his mixes resulting in higher cement content, hence causing a higher drying shrinkage during the setting and hardening of the composite. Zhang et al. (2009) showed that for normal concrete, the ultimate drying shrinkage strain is between 400 \(\mu\varepsilon\) to 600 \(\mu\varepsilon\) whereas typical ECCs, under similar drying conditions (60 % relative humidity and 20 °C) exhibit shrinkage strains of 1200 \(\mu\varepsilon\) to 1800 \(\mu\varepsilon\). The study also suggested the use of a new class of cementitious matrix with characteristics of low drying shrinkage for reducing the shrinkage strain to 109-242 \(\mu\varepsilon\). Compared with PC, this cementitious matrix contains more aluminium oxide and less calcium oxide (Zhang et al., 2009), the latter having the highest thermal expansion and would explain the reduction of shrinkage when used in a lower quantity. However, this type of
laboratory-made cement would be relatively expensive compared with PC and hence its use in large quantities in civil engineering application is not preferred. Mokarem et al. (2003) concluded that mixtures, including ECCs, containing fly ash exhibit a greater drying shrinkage than those containing micro-silica and slag cement, hence suggesting that the latter constituents could help in reducing drying shrinkage. Wang and Li (2006) state that even if the drying shrinkage of ECC is higher than that of normal concrete, it does not necessarily lead to large shrinkage crack widths, due to the pseudo-ductility of ECCs and the ability of the material to support multiple cracking.

2.7.4 Autogenous shrinkage

Autogenous shrinkage is the reduction of volume (macroscopic volume change) due to hydration of cement without any moisture transferred to the surrounding environment (Holt, 2001). In the literature (Holt and Jansen, 1998), autogenous shrinkage is commonly associated with chemical shrinkage (a reduction in volume due to chemical reactions), but occurring at a later stage of the life of the cementitious material.

Autogenous shrinkage is the consequence of withdrawal of water from the capillary pores by the hydration of the unhydrated cement, a process known as self-desiccation (Neville, 2000). In a schematic depiction of a sealed concrete specimen (the surface has been sealed in order to prevent evaporation of water and hence plastic and drying shrinkage), during the hydration of cement (Figure 2-28), Holt (2001) demonstrated how autogenous shrinkage could be part of a larger process of chemical shrinkage. However, chemical shrinkage would lead to an internal volume change while autogenous shrinkage is an external change. Figure 2-28 also shows the possibility of measuring the autogenous shrinkage as a linear change. The parameters influencing the autogenous shrinkage are often disputed, however, it is commonly agreed that it can be controlled through the composition of the mixture (Holt, 2001).
Experiments carried out to evaluate chemical and autogenous shrinkage on cement paste (Hammer, 1999) showed that five hours after mixing, chemical shrinkage could not be integrated with autogenous shrinkage; the latter remained constant after this point while the former continued to increase (Figure 2.29). Practically, this could make the evaluation of these two types of shrinkage more difficult.

Holt (2001) states that an estimation of chemical shrinkage can be made based on the volume of initial and final products, both of which are hardly known:

\[
CS = \frac{(V_c + V_w) - V_{hy}}{V_{ci} + V_{wi}} \times 100
\]  

(2.54)

where \(CS\) is the chemical shrinkage, \(V_{ci}\) is the volume of cement before mixing, \(V_{hy}\) is the volume of hydrated products, \(V_w\) is the volume of reacted water, \(V_c\) is the volume of hydrated cement and \(V_{wi}\) is the volume of water before mixing.
In order to determine the autogenous shrinkage, Loukili (2000) suggests measuring variations of the buoyancy of the specimen from very early ages, where the external volume change caused by cementitious reaction is measured by hydrostatic weighing. As a comparison, a value of autogenous shrinkage for a typical concrete is 40 µε after one month (Neville, 2000).

Normally, autogenous shrinkage would be considered insignificant as the drying shrinkage is usually much larger. However, Neville (2000) states that autogenous shrinkage is particularly high at low water/cement ratios: it is estimated to reach a value of 700 µε for a concrete material with a water/cement ratio of 0.17, whereas for more typical concretes, values of autogenous shrinkage are about 40 µε and 100 µε after one month and five years respectively. This type of shrinkage also tends to increase at high temperatures. Therefore, HPFRCC in general and ECC mixes with a slightly higher cement content in particular compared with a typical concrete are likely to exhibit more significant autogenous shrinkage.

2.7.5 Carbonation shrinkage
Carbonation shrinkage is caused by the dissolving of Ca(OH)$_2$ within the hardened cement (under a compressive stress imposed by the drying shrinkage) which reacts with carbon...
dioxide present in the air to form calcium carbonate $\text{CaCO}_3$, which is then deposited in spaces free from stress (Neville, 2000):

$$CO_2 + Ca(OH)_2 \rightarrow CaCO_3 + H_2O$$ (2.55)

This type of shrinkage is usually limited to the free surfaces of concretes and usually occurs later in the hardened concrete (Dilger and Wang, 1997). The carbonation depth increases with time and the depth of carbonation achieved is proportional to the square root of time (Parrott, 1987).

The decomposition of calcium hydroxide in the cement paste happens mainly at the surface of the cementitious material and in rare cases within the material, when carbon dioxide is present in the hydrated cement paste. However, depending on the environment and quality of the material, carbon dioxide can penetrate beyond the surface of concrete via the pores causing further decomposition of the calcium compound. The rate of penetration of $CO_2$ is dependent on, among other factors, the concrete relative humidity and density (Lamond and Pielert, 2006). Carbonation is accompanied by a slight shrinkage, possibly due to the low volume of the new product formed compared with the volume of the product it is replacing, i.e. the dissolution of calcium hydroxide crystals and deposition of calcium carbonate in its place. Where ECCs are used in hydraulic tunnels with a relative humidity close to saturation, carbonation shrinkage is unlikely to be an issue.

### 2.7.6 Parameters influencing shrinkage

#### 2.7.6.1 Water/cement ratio

Shrinkage and particularly drying shrinkage, defined as the evaporation of water, is significantly influenced by the amount of water present. Neville (2000) states that shrinkage is larger with a higher water/cement ratio which determines the amount of evaporable water and the rate at which water can move towards the surface of the specimen. In an experiment on mortar and concrete specimens stored at a temperature of 21 °C and a relative humidity of 50 %, for an aggregate/cement ratio of 7, a shrinkage value of 200 µε was noted for a water/cement ratio of 0.4 whereas a value of 500 µε was achieved for a water/cement ratio of 0.7.
2.7.6.2 Aggregate/cement ratio

As discussed earlier, aggregates may influence shrinkage of the cementitious material. Zhang et al. (2009) showed that for normal concrete, the ultimate drying shrinkage strain is between 400 \(\mu\varepsilon\) to 600 \(\mu\varepsilon\), whereas typical ECCs, under similar drying conditions (60 % relative humidity and 20 °C) exhibit shrinkage strains of 1200 \(\mu\varepsilon\) to 1800 \(\mu\varepsilon\). Zhang et al. (2009) varying the sand/cement ratios for particular cementitious mixes showed that the shrinkage strain reduces with increasing sand/cement ratio, which is consistent with the behaviour of concrete (Neville, 2000). However, extreme care should be taken for materials containing fibres such as ECC, as a significant content of sand may prevent the fibres from bridging the cracks; the fibres are not able to go through sand particles (Zhang et al, 2009), hence reducing the pseudo-ductility of ECCs.

2.7.6.3 Additives

The shrinkage of cementitious materials can be controlled and even reduced by the use of a range of additives, e.g. metakaolin (Brooks and Megat Johari, 2001), saturated lightweight aggregates (pre-soaked aggregates acting as internal water reservoirs; Sahmaran et al., 2009) and ground granulated blast furnace slag (Lee et al., 2006). However, some of these additives are likely to have a negative impact on the mechanical performance and make the ECCs more brittle. For example, the addition of lightweight aggregates can reduce the tensile load capacity (Sahmaran et al., 2009).

Powder micro-silica (also named silica fume) is an alternative choice of a shrinkage control additive. Typical powder micro-silica is made of solid spherical particles, usually composed of more than 85 % SiO\(_2\) and some aluminium oxide, iron oxide and alkalis. Publications suggest the use of the silica fume as a cement replacement, and according to a study (Zhang et al., 2003); silica fume would be used at a maximum percentage of 10 % by weight of cement. Maghsoudi and Arabpour Dahouei (2007) investigated the use of nano\(^{15}\)-scale materials on the properties of self-compacting concrete for two mixes: one with micro-silica only and the other, a combination of micro- and nano-silica. The results revealed that both compositions exhibit a low shrinkage in air (measured at 400 \(\mu\varepsilon\) at 91 days with the use of only micro-silica and a value of 350 \(\mu\varepsilon\) when using both micro\(^{16}\)-and nano-silica). At the same time, a

\(^{15}\)Nano-scale materials - materials in the nanometre range \(10^{-9}\) m
\(^{16}\)Micro-silica - silica particles in the micro-metre range \(10^{-6}\) m
compressive strength of 60 MPa at 90 days was measured for specimens cured in both wet and dry conditions, and the use of both micro-and nanosilica showed about 4 % increase in compressive strength; a similar observation was made with the flexural strength. The study confirms the ability of micro-silica to reduce shrinkage, which can also enhance strength of the concrete material, however nano-silica material is relatively expensive and in short supply and therefore its use is not preferred, especially as its addition with micro-silica does not make a significant difference in shrinkage and mechanical performance.

Figure 2-30 shows the finer particle size distribution of silica fume compared with PC and other components constituting ECC materials, hence it is probable that micro-silica powder locates itself in-between the other particles constituting the ECC material whilst the material is in the plastic state, hence preventing the evaporation of water and reducing the drying shrinkage of the material. However, according to Sellevold (1987), the use of micro-silica at a high replacement level would increase the autogenous shrinkage (often associated with drying shrinkage), and this is probably due to the refinement of the pore size distribution, which could lead to capillary tension and contraction in the cementitious material. This suggests that a certain limitation of the micro-silica content is necessary.

![Particle size distribution](image)

**Figure 2-30:** Relative comparison of particle size distribution of silica fume with Portland Cement and other components (Fidjestol and Dastol, 2008)
More specifically, Siddique (2011) characterised micro-silica powder as a pore-size refiner (making the pores smaller, hence densifying the matrix, which becomes less permeable), and also as a refiner of the interface between cement and the aggregates. Euclid Chemical Company (2012) also mentioned that micro-silica would react with the Ca(OH)\textsubscript{2} of cement to produce a calcium silicate hydrate gel that enhances the durability and strength of the material. The product is also said to enhance the flexural and compressive strength. Euclid Chemical Company (2012) suggests the addition of powder micro-silica to the cement before incorporating water in the mixture, which is hardly feasible for ECC; indeed due to the high surface area of the micro-silica, water may tend to react with micro-silica instead of hydrating the cement particles. Hence, it would be advantageous when manufacturing ECC material to add micro-silica powder after water addition and only once the water has hydrated most of the cement present.

Embacher (2001) states the benefits of using powder-micro silica as a way of improving the durability of surfaces. Indeed, powder micro-silica, thanks to its fineness, has the ability to fill the voids of the cement structure promoting a dense matrix (Figure 2.31). Figure 2-31 shows the location of micro-silica in-between cement grains. This would make the material more resistant to the penetration of aggressive agents or any fluidic material, as well as increasing the strength of the material.

Figure 2-32 presents the appearance of typical silica fume particles added to a cement paste. The image, where an agglomerate of particles is visible, illustrates how including micro-silica in the manufacturing process of ECC materials can be challenging and can alter it. An additional quantity of “mixing energy” (and probably, an increase in the mixing time) would be required to ensure that the silica fume is well dispersed within the fresh ECC mixture. This would be more important when the “densified” form of micro-silica is used as opposed to the “slurry” form. Silica fume seems to have an effect on the rheology of the cementitious materials. A publication on the effect of silica fume on the rheology of cement confirmed the decrease in workability with the addition of a regular type of silica fume (Kubens et al., 2008). The addition of powder micro-silica reduces the workability of the mixture, increasing the water demand, which has to be taken into consideration when designing the ECC material. Kubens et al. (2008) have also suggested that a better workability is achieved if a high purity silica fume is used instead of standard silica fume.
Figure 2-31: Refinement of pores structure with micro-silica (Embacher, 2001)

Figure 2-32: SEM image of powder micro-silica particles (Fidjestol and Dastol, 2008)
2.8 Summary

This chapter has presented a review of Engineered Cement Composites (ECCs), including their potential use as liners for hydraulic tunnels, both for new construction and for refurbishment of existing infrastructures. ECCs (using polymeric fibres) have an advantage over conventional steel fibre reinforced cementitious material (particularly in the context of hydraulic tunnels) as they prevent the risk of corrosion. The required properties in relation with their intended application are the pseudo-ductility under tensile stress, a long-term durability and a reduced shrinkage.

Having identified the defining characteristics of ECCs compared with fibre reinforced cements, the most important feature of ECCs was described i.e. the ability to exhibit multiple cracks and hence to present a pseudo-ductility under tensile stress whilst achieving a high strain capacity. This is achieved through control of the fibre-matrix interface. The most important aspect of the literature studied is that previous work has focussed on the testing of comparatively thin specimens (13 mm thick) whereas for future civil engineering application, thicker sections must be tested and understood. The parameters controlling the mechanical performance of the material are relatively well understood and include the fibre type and volume fraction, fibre aspect ratio, fibre dispersion and orientation, fibre surface coating, fibre-matrix interface, density and porosity and the addition of controlled defects, but all these factors can be influenced by specimen size.

Cementitious materials could degrade with time due to external factors, however their durability is important. But also, ageing of the cementitious matrix increasing its strength with time may disrupt the balance of properties between the fibre and cement and hence affect the pseudo-ductility of the material with time. The long-term durability related to the fibre-cement interface is not yet certain, however tests conducted on fibres taken from aged specimens, revealed high performance up to 18 years. The literature review also showed evidence of the self-healing ability of the material after the appearance of cracks. Further research in the mechanism of degradation of the material is required and particularly, the ageing process at the fibre-cement interface.

Different types of shrinkage have been identified, in which drying shrinkage is considered as
the most detrimental factor, especially for ECCs in tunnel linings, as it is associated with the formation of cracks and deterioration of structures. Parameters influencing shrinkage such as water/cement ratio, aggregate/cement ratio and additives are of importance. Different additives reducing shrinkage were suggested, including micro-silica in relatively small amounts.

It is therefore clear that there are three areas to be addressed in the current work:

- Mechanical properties of large sections
- Material durability
- Shrinkage on curing

In the next chapter, the experimental methods used to assess these properties are described.
3 Materials and Methodology

3.1 Introduction

In Chapter 2, a class of materials known as Engineered Cement Composites (ECCs) was introduced, and their potential application, particularly with respect to tunnel linings, was discussed. In this chapter, the materials and experimental methods used in the current work are described. As noted in the literature review, the compositions of the ECCs, as well as the mixing procedure, determine the distribution of the constituents in the final composite, which can have an important effect on the performance of the material when measured in both the fresh and hardened state. Hence, the constituents of ECCs and the mixing procedures employed are described in this chapter (sections 3.2 and 3.3).

As discussed in Chapter 2, there are three key areas to consider: mechanical properties, durability and shrinkage. The methodologies for the experimental programmes for these areas are presented in Section 3.4 – 3.7. The methodologies for the durability programme are split between Sections 3.5 (Material and Microstructure Characterisation) and 3-6 (Characterisation of fibre surface and fibre-cement interface chemistry). Finally, shrinkage is also of interest and is an important parameter to understand and control, if these materials are to be taken out of the laboratory and onto site. This is discussed in section 3.7, in which the methods to evaluate the shrinkage behaviour of prism specimens stored under various humidity conditions and containing additives are described and discussed.

In the section which follows, the materials composing ECCs such as the cementitious matrix, fibres and mix designs are detailed, followed by the production of the specimens. Mechanical testing of the ECC specimens are presented along with the single fibre pull-out test method from the cementitious matrix. The techniques to characterise the materials, microstructure, stability of the fibre surface coating and the chemistry of the fibre-cement interface are described, which are important in the evaluation of the long-term durability of the composite. Finally, shrinkage testing along with the evaluation of the influencing parameters are detailed.
3.2 Materials

3.2.1 Cementitious matrix

The main constituent materials for the ECC matrix used in the present work are Portland Cement (PC) containing fly ash complying with BS EN 197-1 (CEM II/B-V 42.5N) and a crushed stone aggregate with a particle size not exceeding 600 µm. The development of an ECC with appropriate properties in the fresh state required the addition of rheological modifiers and admixtures complying with BS EN 934-2. Subsequent studies to investigate shrinkage of the ECCs employed an additive material, micro-silica, in accordance with BS EN 13263-1. Further details on the use of the additive are given in section 3.7, which deals with shrinkage.

Regarding health and safety concerns, Portland fly-ash cement is considered as an irritant (Lafarge, 2011), however all the constituents have been handled and stored following the appropriate Control of Substances Hazardous to Health (CoSHH) regulations. As micro-silica powder is 100 times finer than cement, additional considerations as to the transport, storage and handling must be followed (Concrete Society Working Party, 1993). Micro-silica presents health risks due to the fineness of the particles and CoSHH regulations have been followed, especially during batching and subsequent use where it advised to avoid long-term exposure and to use protective equipment such as dust masks as well as sufficient mechanical ventilation. Hence, extreme care should be taken when mixing the ECC components and preparing the specimens for testing.

3.2.2 Fibres

As discussed in Chapter 2, Polyvinylalcohol (PVA) fibres are a comparatively new reinforcing material for the cement industry. Their yield strength is reported to be higher than structural steel and they have a high strain at failure (see section 2.3). In the current study, PVA fibres that are ‘tailored’ for use (in that a surface coating is applied to control the bond-strength between the fibre and the matrix) as the reinforcement in ECCs have been used. The fibres have a nominal diameter of 40 µm and a length of 8 mm. Two types were investigated: Type 1 (T1) and Type 2 (T2).

The key difference is that T2 is the resin-bundled version of T1. There is an additional stage in the manufacturing process that holds the fibres together in a bundle. Data from the supplier (Kuraray) indicates that the Young’s modulus and tensile strength of the PVA fibre are 40 GPa...
and 1560 MPa respectively. Tensile testing conducted on the same PVA fibres confirmed similar values of Young’s modulus and tensile strength (Magalhaes et al., 2013).

As discussed in section 2.3.4, the optimum fibre content to observe multiple cracking behaviour is 2 % by volume (Li, 2002b): a fibre content lower than 1.5 % causes the ECC not to show the desired multiple cracking behaviour, while a fibre content higher than 2.5 % reduces the workability of the fresh mix and increases the cost.

PVA fibres are not considered as hazardous or harmful (Kuraray Co. Ltd, 1999), although it is advised to wear a dust mask, safety gloves and goggles when handling the fibres due to the size of the particles involved. The fibres are considered as a safe material as it is composed of carbon, hydrogen and oxygen. Hence, when burning; only H₂O and CO₂ are released, with no other harmful substances produced (Kuraray Co. Ltd, 1999).

### 3.2.3 Mix Designs

Three mixes are considered (Table 3-1). Composition A was made of Portland cement (PC) containing fly ash, fine aggregates, water, admixtures and PVA fibres. A polymer additive (based on a copolymer of ethylene-vinyl acetate) was included in some mixes to help control shrinkage, and hence strength and durability (composition B), whilst powder micro-silica (mean diameter 0.15 µm), was incorporated in composition C. Figure 3-1 illustrates the powder micro-silica particle size distribution (in volume and cumulative content percentages), as provided by the supplier (90 % of the particles have a diameter below 0.573 µm whilst 10 % of the particle sizes are below 0.069 µm). As detailed in section 2.7.6.3, micro-silica particles tend to agglomerate, hence the mixing procedure should ensure its proper dispersion and due to the high surface area, water is added to the mixture before the micro-silica to ensure that the water is used to hydrate the cement particles first.
### Table 3-1: ECC mix design (ratios given are by mass)

<table>
<thead>
<tr>
<th>Constituent details</th>
<th>Compositions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
</tr>
<tr>
<td>Portland cement (c)</td>
<td></td>
</tr>
<tr>
<td>Fine aggregates (a)</td>
<td>a/c = 0.68</td>
</tr>
<tr>
<td>Water (w)</td>
<td>w/c = 0.34</td>
</tr>
<tr>
<td>Admixtures (ad)</td>
<td>ad/c = 0.006</td>
</tr>
<tr>
<td>PVA fibres (f)</td>
<td>2 % vol.</td>
</tr>
<tr>
<td>Polymer (p)</td>
<td>/</td>
</tr>
<tr>
<td>Micro-silica (ms)</td>
<td>/</td>
</tr>
</tbody>
</table>

![Figure 3-1: Micro-silica particle size distribution data provided by the supplier (Elkem, 2012)](image)

### 3.3 Specimen Production

#### 3.3.1 Mixing process

The mixing process specifies the way the different constituents are combined: this includes the order of addition and the mixing time. As discussed in the literature review, this can have a significant effect on the spatial distribution of the different components in the ECC and therefore on the performance of the composite in the fresh and hardened state. This is because the mixing method, and order of mix addition influence the workability of the mixture. As a result, in addition to the fibre type, two processes were investigated in order to determine the impact on:

a) the workability of the fresh ECC mix and the ease with which it could be cast into prepared metal moulds;
b) the distribution and dispersion of the PVA fibres within the cement mortar matrix; and  
c) the size, scale and distribution of any entrapped air (porosity) within the ECC.

The point in the manufacturing cycle when the fibres are added is likely to have an effect on the eventual distribution of the fibres in the cured ECC:

- In Process 1 (P1), the PVA fibres were added to the dry ingredients prior to the addition of water
- Process 2 (P2), water was added to the mix before the fibres.

It should be noted that Kuraray recommends only Process P1 in conjunction with T2 fibres. This was borne out in the initial testing which showed that the dispersion of the resin bundled fibres was very poor with Process P2. Under these circumstances, this combination was not considered further.

The constituents of each batch of ECC were mixed using either a Hobart commercial small scale mixer (6 litre-capacity), Figure 3-2a, or a conventional concrete mixer for larger mix volume, Figure 3-2b. The different components were added successively, mixing until a homogeneous distribution was achieved before adding the next component.

In order to deploy ECCs in real engineering situations (e.g. tunnel lining applications), there is a need to understand the workability of the mix, i.e the associated ease of placement and resistance to segregation. Hence the workability of the mixes produced was measured using a flow table following BS EN 1015-3. In this method, the fresh mixture is spread on a disc and
the extent of spread is used to indicate the workability. The workability of the mixture was established during the development of the product and a target workability of 165 mm ± 15 mm is desirable for the application of ECCs in tunnelling applications whilst preserving the self-compactability of the mixture (Psomas and Eddie, 2009).

3.3.2 Casting procedure
As discussed in section 2.5.3.4, a previous study conducted on polypropylene fibres in a cementitious matrix demonstrated a clear link between the casting method and the mechanical performance (Takashima et al., 2003). This was attributed to the casting procedure having an effect on the fibre orientation in the resulting samples.

In this programme, the fresh mixture was poured into a mould to produce dog-bone shaped specimens 30 mm in thickness (Figure 3-3.a). As a comparison, slabs (1000 mm x 800 mm x 30 mm, i.e. of a similar thickness to the dog-bones) were also cast and from which dog-bone specimens were subsequently cut. The influence of the casting process on the mechanical properties of ECC samples was investigated by comparing the performance of 30 mm thick “dog-bone” specimens (as described in section 1.3) cast into shaped metal moulds (Figure 3-3.a) with equivalent specimens cut from a slab (1000 mm x 800 mm x 30 mm) of ECC material using an abrasive water jet cutting process, Figure 3-3.b. The water-jet cutter was a Global Cutting Technologies, YC-L1212 using Naiky, NcStudioTM-V9 control software; the cutting parameters used were 150 mm/min and 100 Mesh Garnet sand.

Figure 3-3: Specimens 30 mm thick (a) manufactured in a mould (moulded) and (b) cut from a slab using a water-jet cutter (machined)
3.4 Mechanical testing

3.4.1 Introduction

The key feature of ECC, when compared to conventional cement mortars and concrete, is its ability to exhibit pseudo-ductility when tested in tension, which means that the onset of cracking does not necessarily signify catastrophic failure. In properly formulated ECC, catastrophic fracture should happen only at much higher strains (Li et al., 2001). Consequently, while (cube) compression tests have been carried out on the ECCs studied here, the resulting values are not particularly helpful in evaluating potential structural performance, since they provide no indication of failure mechanisms in tension. Therefore, tensile and flexure testing were considered more appropriate when assessing the performance of the ECCs. The test methods used and their relation to various standards are discussed below.

ECCs are a relatively new material and whilst there is a growing body of literature, there are, as yet, few specific standards for the testing of these materials. Previous work carried out in Japan on High Performance Fibre Reinforced Cementitious Composite (HPFRCC) materials has led to the development of some standard test methods. Since ECCs represent a development of this class of materials, it seems sensible to use this standard as a starting point (The Japanese Society of Civil Engineers, 2008). The British standards for testing concrete (and metallic fibre reinforced concrete) have also been used when testing the specimens in flexure.

3.4.2 Tensile Testing

3.4.2.1 Introduction

From an engineering perspective, the fundamental attraction of ECCs is pseudo-ductility, which means that structural failure by catastrophic fracture is less likely to happen. Tensile testing is the most severe mode of loading for the material, whereas flexure is more likely to promote a graceful failure. Consequently, the performance of the ECC in tension is critical and has been used in the current work, notwithstanding the inherent challenges in testing a brittle material in tension.

Within the scope of this study of the properties and performance of ECC, tensile testing was used to evaluate, a) the effect of different constituents such as fibre type and processes, and b) the ability of the material to ‘heal’ autogenously.
3.4.2.2 Japanese Standard and Original Geometry

As noted in Section 2.5.2, previous studies have focussed on thin specimens and the “Japanese standard” (JSCE, 2008). Initially developed for High Performance Fibre Reinforced Cement Composites (ECCs being a development example on this type of material), the standard specifies the use of thin dog-bone shape specimens 13 mm thick (Figure 3-4a), with an angled shoulder between the ends (with a width of 60 mm) and over the 80 mm gauge length having a width of 30 mm. Whilst thicker sections have been tested (Kanakubo, 2006; Kim et al., 2007), further work is still required to understand thickness effects and the effect of fibre orientation on strength. In the current study, the specimen thickness has been increased to 30 mm. Therefore, the specimen thickness is almost four times the fibre length, which is 8 mm, compared with thinner sections where the fibre length is closer to the actual thickness.

Initially, tensile testing was carried out using a geometry based on the dog-bone of the Japanese standard (Figure 3-4a), but with a specimen thickness of 30 mm (Figure 3-4b). Hence, when casting ECCs in the thicker mould, it is more likely that fibres will be randomly distributed in all directions and this is likely to affect the mechanical performance of the ECC loaded in tension.

![Figure 3-4: (a) Schematic of unconfined tensile test as illustrated in the Japanese Standard after JSCE, 2008 and (b) Original dog-bone geometry adapted from (a) and as tested initially](image)

Initial results from testing 30 mm thick specimens prepared from Composition A mixes indicated that there was limited pseudo-ductility after the initial crack (Figure 3-5a) when
compared with the results reported in the literature (Li et al., 2001). Further, the specimens always failed at the sharp transition in width at the end of the 30 mm wide test section (Figure 3-5b) and there were only a limited number of cracks observed within the nominal gauge length of the specimen prior to failure. As expected, repeated failure of specimens at the sharp edges and the presence of limited pseudo-ductility confirmed an issue with the geometry of the specimen. The sharp edges were concentrating stresses, preventing extensive multiple cracking within the gauge length of the specimen and hence the expected pseudo-ductile behaviour under stress could not be observed. It was therefore anticipated that the elimination of these sharp transitions would allow the multiple cracking behaviour reported in the literature to occur. As a consequence, a finite element analysis (using Abaqus software) was carried out to verify the hypothesis of the existence of stress concentrations at the sharp edges and to determine the best geometry of the tensile specimen to adopt. Ideally, important tensile stresses should be located within the gauge length of the specimen, where multiple fine cracks are expected to occur progressively.

![Tensile stress vs strain graph](image)
3.4.2.3 Finite Element Analysis

An analysis of the tensile stress distribution within the thick dog-bone specimens under a tensile load of 5 kN (equivalent to a nominal gauge length stress of 3.33 MPa) was conducted using finite element analysis FEA (Abaqus 6.10-1). The model is linear elastic up to the first crack and thereafter perfectly plastic behaviour and where $E = 10$ GPa and $\sigma_y = 4.9$ MPa, using data obtained from testing a specimen in tension.

As expected, FEA confirmed the existence of stress concentrations at the transition from the sharp corner at the shoulder to the gauge length with the original geometry (Figure 3-6.a). Consequently, the geometry was modified by introducing a curved shoulder to the specimen. FEA immediately showed that when the shoulder is curved rather than angled, there is a smoother distribution of the tensile stresses and a reduced concentration of stress (Figure 3-6.b-d). The geometry selected was the one exhibiting least stress concentration at the edges and the development of the highest tensile stresses at the middle of the gauge length of the specimen (Figure 3-6.c); the radius of curvature for this geometry is 185 mm.
Chapter 3 – Materials and Methodology

(a)
Figure 3-6: Abaqus output for dog-bone specimens with varying radii of curvature (R) between the gripped region and the gauge length (a): original specimen, (b) R = 175 mm, (c) R = 185 mm, (d) R = 195 mm. Response indicates the stress distribution (MPa) throughout the specimen for a given load of 5 kN. Blue indicates the lowest stress values and Red, the highest.
3.4.2.4 Tensile test method

Having modified the test geometry, further tensile testing was carried out to evaluate the effect of process and fibre type on mechanical behaviour. Dog-bone shaped specimens (30 mm thick, Composition A), with the geometry shown in Figure 3-7a, were loaded in tension. The flexibility to accommodate imperfections in the specimen geometry and misalignment in the test machine is provided by the pin situated at the top grip (Figure 3-7b). The top and bottom grips have been designed to fit both the geometry of the specimen and the tensile testing machine. Table 3-2 shows the experimental test matrix relating to the fibre and process. Given previous experience, Process P2 with Fibre T2 was not tested. Three specimens were manufactured per fibre and process type; specimens were tested at 27 days.

Table 3-2: Experimental test matrix relating to fibre and process types (LD = Cross-head displacement rate during testing)

<table>
<thead>
<tr>
<th>Fibre Type</th>
<th>Process Type</th>
<th>Process 1 (P1)</th>
<th>Process 2 (P2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type 1 (T1)</td>
<td>LD = 0.05 mm/min</td>
<td>LD = 0.05 mm/min</td>
<td>N/A</td>
</tr>
<tr>
<td>Type 2 (T2)</td>
<td>LD = 0.05 mm/min</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

Specimens were loaded in tension, using a Universal Testing Machine (Instron 4505 5500R, with a load cell of 10 kN capacity and using Bluehill 2, Instron’s proprietary software for control and data acquisition) in displacement control mode at a rate of 0.05 mm/min. A slow rate was chosen in order to enable the detection of most of the events occurring when the specimen was tested in tension, such as the formation of multiple fine cracks. The Bluehill 2 software enables the load to be recorded as a function of the crosshead-displacement of the machine for all specimens. Using this test method, the strain to first cracking derived from the crosshead displacement is not accurate, due to the effect of load train compliance, but because the subsequent multiple cracking occurs at a near constant stress, the measurement of strain capacity after the first crack is accurate. In order to get the correct displacement (strain), associated with the gauge extension length of the specimen only, some specimens were instrumented on the front face with a Linear Variable Differential Transformer (LVDT, Figure 3-8a) or with strain gauges (one on each side) optimised for use with concrete (TechniMeasure, type PL-60-11; Figure 3-8b). The LVDT was calibrated using a matrix gauge block and a LVDT calibration device based on a micro-meter (Figure 3-9). Values of strain
obtained from the cross-head displacement of the machine, LVDT and strain gauges were compared to determine the most sensible measure of strain as a function of stress in order to determine the Young’s modulus (E), which is an important parameter for the design of structures.

Figure 3-7: Tensile test arrangement for (a) New test specimen geometry and (b) Gripping arrangement

Figure 3-8: Revised test geometry (a) with LVDT on front face and (b) with strain gauge (optimised for use with concrete specimens, 60 mm in length mounted on the side of the specimen)
Some older specimens were also tested and in addition, the ability of ECCs to self-heal was investigated following a specific experimental programme as noted below:

- Initially tested in tension at 28 days up to different displacements (0.3 mm and 0.5 mm), cured in water at 20 °C before being re-tested 28 days later (i.e. 56 days in total).
- Initially tested in tension at 28 days up to a 0.5 mm displacement and then re-tested after different curing durations: 14 days, 28 days and 42 days.
- A specimen successively cured in water and tested every month up to 4 months, then at 7 months and finally every month up to the final test at 11 months when it was failed; the specimen is tested during the first 4 months to increasing displacements and then to similar displacements until the final testing, this being the 9th test.

### 3.4.3 Flexural Testing

Flexural testing was also used to determine the mechanical properties of the ECCs. It was used to evaluate, a) the effect of different constituents and processes, b) the effect of aging on mechanical properties and, c) the ability of the material to ‘heal’ autogenously.

Beams (500 mm x 100 mm x 100 mm; Figure 3-10) were loaded in four point bending (4PB) using a Controls, Digital Triaxial tester T400 with a load capacity of 100 kN in displacement control at a rate of 0.2 mm/min. A higher speed was chosen in this case compared to the tensile testing in order to achieve cracking in a comparable time, as the specimens here were larger. The four point bending test was preferred over the three point test as it produces a larger region of uniform stress for the multiple cracking behaviour of the specimen. Figure 3-10 shows the geometry and load application points (BS EN 12390-5: 2000).
During testing, displacement was measured using two LVDTs, fixed on the beam specimen (Figure 3-11). The LVDTs were placed on the same side of the specimen, separated by a distance of $d_0 = 70$ mm. Initial tests were initially carried out with the LVDT placed over a length of 50 mm, following the guidelines in the British Standard for testing metallic fibre reinforced concrete (BS EN 14651: 2005). However, under these conditions, it was observed that the multiple fine cracks often appeared outside the measured region. Therefore, the region that the LVDTs cover was increased to 200 mm, which represents an overlap of 50 mm either side of the upper rollers (Figure 3-11). This is slightly longer than the region which is subject to the greatest bending moment and should therefore account for the entire region where cracks would be expected to occur. As the LVDT spans over 200 mm, whilst the central constant moment area has a length of 100 mm, a part of the LVDT does not experience the same strain field as occurs within the central span, hence the measured values $\varepsilon_t$ and $\varepsilon_c$ are an underestimate and need to be corrected for, as shown schematically in Figure 3-12 and equation 3.2 for $\varepsilon_t$. 

Figure 3-10: Schematic diagram of the flexure test specimen (dimensions in mm)
Figure 3-11: Image showing the new position of the LVDTs on the beam tested in flexure

![Image of LVDTs on beam]

Figure 3-12: Schematic illustrating the calculations for the corrected strain $\varepsilon_{t,m}$

\[
\varepsilon_t = \frac{100}{200} \varepsilon_{t,m} + \frac{100}{200} \frac{3}{4} \varepsilon_{t,m} = \frac{7}{8} \varepsilon_{t,m} \tag{3.1}
\]

Hence:

\[
\varepsilon_{t,m} = \frac{8}{7} \varepsilon_t \tag{3.2}
\]

Calculations using the data obtained have been carried out based on the standard for testing hardened concrete (BS EN 12390-5:2009) and the published Japanese concrete standards (JCI-S-003-2007). Load and strain data were used to produce Moment-Curvature plots.

The flexural strength was calculated using the following equation from BS EN 12390-5: 2009:

\[
\sigma_{cf} = \frac{F_{max} l}{d_1 d_2^2} \tag{3.3}
\]

where $F_{max}$ is the maximum flexural load, $l$ is the distance between the supporting rollers (i.e. the span equal to 300 mm), $d_1$ and $d_2$ are the lateral dimensions of the specimen.
The bending moment $M$ was calculated using the following equation from JCI-S-003-2007:

$$M = \frac{F \cdot l}{2}$$  \hspace{1cm} (3.4)

where $F$ is the applied load and $l$ is the span (= 300 mm).

The curvature $\phi$ gives a measure of the degree of (uniform) bending in the sample and may be determined using equation 3.5:

$$\phi = \frac{\varepsilon_t - \varepsilon_c}{d_0}$$  \hspace{1cm} (3.5)

where $\varepsilon_t$ and $\varepsilon_c$ are the surface strains in tension and compression respectively and $d_0$ is the distance between the two LVDTs. The corrected value of the strains $\varepsilon_{t,m}$ and $\varepsilon_{c,m}$ are used in equation 3.5.

As a comparison with the tensile stress, the bending stress will be estimated. The bending stress at first crack can be calculated from the bending moment at first crack $M_{fc}$ using the following equation:

$$\sigma_{b,fc} = \frac{M_{fc} \cdot y}{l}$$  \hspace{1cm} (3.6)

where:

$$l = \frac{d_1 d_2^3}{12}$$  \hspace{1cm} (3.7)

And

$$y = \frac{d_2}{2}$$  \hspace{1cm} (3.8)

using $d_1 = 100$ mm and $d_2 = 100$ mm corresponding to the lateral dimensions of the beam specimen.
3.4.4 Single fibre pull-out

In this work, the single-fibre pull-out (SFPO) test was used to investigate the fibre-cement interface behaviour. The test was also carried out on specimens of different ages in order to contribute towards the understanding of long-term durability of ECCs. Single fibre pull-out testing was carried out in collaboration with the Leibniz Institute of Polymer Research (Leibniz-Institute für Polymerforschung) in Dresden, Germany. The Institute possesses specialist equipment enabling micromechanical tests in different loading modes (Leibniz Institute for Polymerforschung, 2012). Here, a quasi-static pull-out test method was chosen; the development of the apparatus is described by Mäder et al. (1994).

On the basis of the mechanical testing programme results, only T1 fibres (non-resin bundled) were used for the SFPO tests. This fibre type led to enhanced mechanical performance of the ECC. The cementitious matrix was made of Portland cement, admixtures and water at w/c ratio of 0.34 (see section 3.2.3) as in composition A, but without the aggregate content. The fresh cementitious matrix is very fluid and therefore it was decided to wait one hour before inserting the fibres. This decision was not expected to have a significant effect on the results beyond ensuring that the test specimen could be properly processed. The single fibres were embedded in the cementitious matrix using an average length of $883 \pm 246 \, \mu m$. After 24 hours, the specimens were transferred to a desiccator and left to cure in a relative humidity environment greater than 90 % for different lengths of time. The quasi-static pull-out tests were carried out at a speed of $0.01 \, \mu m/s$ initially and then switched to $1 \, \mu m/s$ at a displacement of $10 \, \mu m$ subsequent to the maximum force being reached.

Tests were carried out in batches after 7 days, 28 days, 3 months and 5 months. SFPO tests tend to show some variability and hence for statistical reasons and a better estimation of the interfacial parameters, a minimum of 20 tests were carried out for each age.

3.5 Material and Microstructural Characterisation

3.5.1 Introduction

A number of parameters can influence the mechanical properties of ECC and indeed the long-term durability. The manufacturing process can potentially lead to preferential fibre alignment, clustering of pores and variation in pore size. Further, whilst it is hoped that there
will be unreacted cement present, which will promote the autogenous healing ability of the material, this is also influenced by the crack widths, which need to be kept to a minimum.

Density and porosity as well as fibre distribution and orientation have been measured and characterised. Fractography and the analysis of the fibres pulled-out in the single fibre pull-out test have also been carried out, which in addition to supporting the work on fibre distribution has contributed to the understanding of the failure mechanisms at work. The presence of un-reacted cement was characterised as well as were the possible healed products formed. The crack widths have also been measured. In the present section, a range of techniques that can be used to analyse these phenomena are discussed.

3.5.2 Density and Porosity measurements
Density and porosity measurements were made using samples cut from both tensile and flexural specimens, enabling a direct correlation of the mechanical performance with the physical properties.

The density of the samples was measured according to the method described in BS 12390-7: 2009. The water saturated surface-dry samples were weighed in air to determine their mass ($W_s$). The volume $V$ they displaced once in water was measured using a water displacement technique.

In the absence of any British Standards for evaluating the porosity of concrete specimens, the starting point for the present work was a study comparing the ASTM saturation techniques for measuring the permeable porosity of concrete (Safiuddin and Hearn, 2005). This paper recommended the use of the vacuum saturation technique as the most efficient method. The study confirmed the efficiency of the vacuum saturation technique over the cold-water or boiled-water saturation technique. Indeed, the vacuum saturation method would provide the highest permeable porosity. Based on this, ASTM 1202-97 was followed to measure the porosity of the ECCs.

Porosity was determined as follows. The samples were placed in the oven for 24 hours at 50 °C to dry and then weighed to determine the oven dry mass in air ($W_d$). Samples were then placed under vacuum to eliminate as much of the air present as possible prior to the introduction of
water, which occupies the evacuated volume. The samples were weighed again to determine
the buoyant mass of the specimen in water ($W_b$). The permeable porosity (PP) is then given
by equation 3.9 (e.g. Safiuddin and Hearn, 2005):

$$PP\, (\%) = \frac{(W_s - W_d)}{(W_s - W_b)} \times 100$$  \hspace{1cm} (3.9)

### 3.5.3 Scanning Electron Microscopy

#### 3.5.3.1 Introduction
The microstructure of the ECC was analysed using a Hitachi S3200N Scanning Electron
Microscope (SEM). Different modes of the SEM were used:

- The secondary electron mode was used to examine features at high resolution and
  precision. This mode of the SEM was used to examine fracture surfaces from the
  specimens tested in tension (and containing fibres) and aspects of the fibres pulled
  from the cementitious matrix from the single fibre pull-out tests.

- The backscattered electron mode was used to analyse the fibre dispersion and
  orientation, and to estimate the composition ratio of the different components of the
  ECC, including unreacted cement.

- The energy-dispersive X-rays mode (EDS-SEM) was used to determine the chemistry of
  the possible healing products forming the healed cracks inspected.

#### 3.5.3.2 Fractography and fibres from the single fibre pull-out tests
In the secondary electron mode of the SEM, the source of electron is usually of tungsten
filament and the electrons are accelerated to an energy usually between 1 keV and 30 keV
(Goodhew, Humphreys and Beanland, 2000). The high energy electron beam scanning the
surface of the sample produces images with an excellent spatial resolution in the nanometre
range (10 to 50 nm), which is ideal for the current fibres having a diameter of 40 µm. In
addition, the large depth of field is an important aspect of the technique, giving information
about the topography of the sample (Goodhew, Humphreys and Beanland, 2000). However,
the technique is limited by the existence of charging effects at high accelerated voltages (due
to the build-up of electrons at the surface of the specimen analysed) making the image
extremely bright. Rice (2009) discusses different types of charging effects that can be present,
such as edge charging (where electrons build up on high or isolated portions of the sample),
area charging (where electrons charge and discharge in certain areas), line by line charging (where electrons are released randomly from the sample line by line, causing very bright streaks across the image) and residual charging (where the electrons left from a previous scan are added to the current ones). He also mentioned that the more non-conductive a sample is, the more charging becomes a problem. Possible solutions include using a low accelerating voltage, reducing the spot size (this reduces the number of electrons hitting the sample), lowering the vacuum where the gas in the chamber absorbs some excess of electrons (also known as environmental scanning electron microscopy) or coating the sample with a conductive layer. However the use of these techniques can compromise the achievable resolution of the instrument.

Small samples were cut from the cracked faces of the tensile specimens and gold coated (four coatings of 99.9 % gold each 2 nm thick) to make these conductive for SEM examination and to avoid charging effects. The examination of the fibres from the fracture surface was used to obtain evidence relating to the failure mechanisms: whether the fibres pulled-out or fractured in the cementitious matrix. In addition, the matrix porosity could be visualized and preliminary observations regarding the fibre distribution and the fibre-matrix interface could be made.

Similarly, individual fibres from the single fibre pull-out tests have been gold coated and examined using the secondary electron mode of the SEM. The observations have been correlated with measurements of load versus displacement obtained during single fibre pull-out testing (Figure 3-13), in order to relate the physical aspect of the fibre with its mechanical behavior in tension and within the cementitious matrix.
3.5.3.3 Fibre orientation and dispersion, and unreacted cement

The backscattered electron mode of the SEM enables the visualisation of some features of a material based on a Z-contrast (the lower the atomic number the darker the image appears, whilst conversely the higher the atomic number, the lighter the image). This ‘Z contrast’ method enables the determination of the different elements present in an image (Lloyd, 1987). This mode of the SEM was used to visualise the fibre orientation of the specimens tested in tension, the fibre dispersion and the presence of unreacted cement. The fibres are recognised by their dark colour (relatively low Z as mainly composed of carbon), their size and shape, whilst the unreacted cement is lighter compared with its surroundings.

The fibre orientation was analysed by examining samples (50 mm x 30 mm in area) cut from near the fracture surface of the specimens tested in tension. The examination of the fibre dispersion and orientation requires a specific sample preparation for the fibres to be identified easily. Further, the identification and quantification of various phases of the cementitious matrix require the sample to be prepared well and usually a flat surface is preferred (Kjellsen et al., 2003). Several stages were required to obtain a perfectly flat and smooth surface for

Figure 3-13: Image showing the fibres fixed individually on a purpose-built device after the single fibre pull-out test, ready for SEM examination
examination. In the present work, flat and smooth surfaces were prepared as follows:

- The cut sample was placed in a mould (such that the cut face could be polished), then vacuum-filled with an epoxy resin, which also filled the pores of the material. The resin stabilises and maintains the microstructure, enabling the sample to withstand grinding and polishing without alteration. It prevents particles plucking, pitting and micro-cracking the polished surface.

- After curing the epoxy overnight, samples were submitted to a series of grinding and polishing steps with progressively finer abrasives following the schedule presented in Table 3-3 (which was developed following Kjellsen et al., 2003), in order to produce a flat polished specimen. A high quality finish is necessary to facilitate the identification and quantification of various phases of the cementitious material (Kjellsen et al., 2003).

- The polished specimens were then sputter coated with a film of conductive material in order to prevent the charging effect when the specimen is scanned by an electron beam. Coatings were either 99.9 % gold (four layers of 2 nm each) or carbon.

### Table 3-3: Grinding and polishing stages (menu)

<table>
<thead>
<tr>
<th>Stages</th>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
<th>VII</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equipment</td>
<td></td>
<td></td>
<td></td>
<td>Planapol</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specimen holder</td>
<td></td>
<td></td>
<td></td>
<td>Pedemax 3 specimens holder</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grinding media</td>
<td>SiC</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Grit/Grain size</td>
<td>81 µm</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Polishing cloth</td>
<td>/</td>
<td>Metal bond</td>
<td>Resin bond</td>
<td>DP-DUR (silk cloth)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Polishing media</td>
<td>/</td>
<td>Diamond pad</td>
<td>Diamond Suspension</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grain (µm)</td>
<td>/</td>
<td>75</td>
<td>20</td>
<td>10</td>
<td>6</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>Lubricant</td>
<td>WATER</td>
<td>BLUE</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Speed (rpm)</td>
<td>300</td>
<td></td>
<td>150</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Time (mins)</td>
<td>2</td>
<td>10</td>
<td>5</td>
<td>2</td>
<td>2</td>
<td>2.5</td>
<td>3</td>
</tr>
</tbody>
</table>

The unhydrated cement in the material and the fibre dispersion were evaluated on specimens made following Composition C, using fibre T2 and Process P1. The specimens analysed were one year old. The presence of un-reacted cement is examined on both specimens placed in water and in air. Similarly to the examination of fibre orientation and dispersion, the visualisation of such features via the backscattered electron mode of the SEM requires specific preparation of the sample, with several stages, in order to make the surface of the sample flat.
and smooth for the analysis. Specimens cut into cubes of approximately 1 cm$^3$ in size, were placed in an epoxy resin followed by several stages of grinding and polishing. The samples were prepared following the procedure presented in Table 3-3. However, ethanol was used as a lubricant instead of water so as to preserve the un-hydrated cement and to obtain a flat-polished specimens. The procedure detailed in Table 3.3 involves a series of grinding and polishing steps with progressively finer abrasives as follows: 3 x 30 seconds with 75 µm, 30 seconds with 20 µm, 30 seconds with 10 µm, 60 seconds with 6 µm, 150 seconds with 3 µm, 180 seconds with 1 µm. The polished specimens were then sputter coated with a film of carbon.

### 3.5.3.4 Chemistry of the healing products

When the Energy Dispersive X-ray Spectroscopy (EDS) mode is used with SEM, it gives information about the chemical composition of the material analysed: both qualitative and quantitative analyses are undertaken. The technique utilises the X-Ray emitted by the specimen when bombarded by the electron beam: the electrons emitted create a vacancy that is filled by an electron from a higher orbital shell, causing the emission of an X-ray due to the difference in energy between the two electrons. The X-ray emitted has a unique characteristic energy and therefore can be used to identify the atom from which it is emitted. The composition can be presented both as a spectrum of relative intensities and as a percentage breakdown of the significant elements. The advantages of this technique over others (Levy, 1989) is that all the elements with a $Z \geq 11$ (Na) can be detected ($Z \geq 6$ (C) with thin detectors), the detection limit is to within 0.1 % by weight, it is a fast method (in terms of data collection) and most importantly rough surfaces seen on the images can be analysed thus adding information to topographic SEM results. The limitation of the technique is that for some combinations of elements, large differences in self-absorption and fluorescence of emitted x-rays will limit the precision of quantitative analysis.

### 3.5.4 Reflected Light microscope equipped with a camera

The reflected light microscope is used to identify and measure the crack widths of the healed specimens. This will give information about the possible autogenous healing ability of the specimen, which is also dependent on the crack width as detailed in section 2.6.5.

The microscope uses visible light which is focussed through the specimen by lenses producing
a magnified image. This technique offers the advantage of direct imaging, a quick and easy access to the specimen to be analysed, without the need of any specific sample preparation. However, the disadvantage of the optical microscope is the low resolution, down to only a sub-micron and the relatively poor depth of field. Here, an Axiophot Microscope (Zeiss, Germany) was used.

3.6 Characterisation of fibre surface and fibre-cement interface

3.6.1 Introduction

As detailed in section 2.6.4, the long-term durability of the composite material is closely linked with the fibre-cement interface, therefore identifying the changes of the fibre surface chemistry and particularly the interface would give valuable information on the maintaining of the pseudo-ductility of the ECCs with time. In this section, the techniques used to determine the changes of the fibre surface chemistry and to evaluate the chemistry of the fibre-cement interface are detailed.

3.6.2 Time of Flight - Secondary Ions Mass Spectrometry (ToF-SIMS)

This technique, which is very surface sensitive, was used to compare the surface chemistry of the fibres kept in air with those kept in water for nearly three years, hence evaluating the stability of the coating after long-term exposure to (tap) water. It is possible that physico-chemical changes to the fibre coating may occur with time, which will affect the maintaining of the pseudo-ductility of the ECC with time. The findings will also be compared with the results of the single fibre pull-out data at different ages to confirm the impact of possible changes with time. This was intended to gather valuable information on the long-term durability of the ECC.

The technique, using a ToF-SIMS 5 (ION-TOF GmbH, Münster, Germany) is used to characterise the composition of surfaces (and also interfaces and bulk profiles). The technique is based on a time-of-flight mass analysis of the secondary ions emitted by a sample when its surface is sputtered with a primary ion beam (in this case Bi$^{3+}$). The sputtering phenomenon is described as a high energy primary ion beam dislodging secondary ions from the atomic layers of the surface of the material. The depth of analysis is in the order of 1-2 atomic layer(s). The technique requires an ultra-high vacuum to increase the mean free path of ions liberated in the flight path. The intensity of the elements detected is plotted against their mass: the mass
spectra are used to determine the elemental and molecular species on a surface. A mass spectrum is generated by measuring the arrival times of the secondary ions at the detector and by performing a time to mass conversion.

The fibres need to be well-separated as they tend to generally stick to each other: a flat surface on the fibres is identified for analysis. A sample sputtered by an ion beam could lead to molecule fragmentation of its surface, the fragmented molecules are ionised and analysed to identify their atomic mass, which are then used to determine the chemistry of the molecules present on the surface.

3.6.3 X-Ray Photoelectron Spectrometry (XPS)

The XPS technique will enable a better understanding of both the stability of the fibre coating and the behaviour of the fibres in cement, both of which could also help the modelling of the fibre-cement interface. With a depth of analysis of up to 10 nm, X-ray Photoelectron Spectroscopy (XPS) is a surface analysis technique which is complimentary to ToF-SIMS. In addition to determining the elemental composition of a sample, the bound state(s) of any elements present can be determined. Here, a Theta Probe (Thermofischer Scientific, East-Grinstead, UK) has been used.

As a monochromated X-ray source (in this case Al Kα) is used, the input energy is of a known quantity. By measuring the kinetic energy of the photo-electrons released, it is possible to determine their bound energy. The technique enables not only the detection of the different atoms present on the fibres but also the chemical bonds present: a slight, but measurable, shift in binding energy is observed when one atom is chemically bound to another. The shift value enables the determination of the binding species of each element. The binding energy of an electron in a particular orbital is unique and so it is possible to differentiate between, for example a Carbon 1s (C1s) and an Oxygen (O1s). Further, when an atom is involved in a covalent or ionic bond, the binding energy of the electron is shifted slightly so it is possible to tell the difference between a C-C bond and a C-O bond, for example. This characterisation technique requires ultra-high vacuum conditions to prevent any interaction of the elements detected with the atmosphere.

Good sample preparation is important to ensure the quality of the data obtained: the surface
of the sample to be analysed should ideally be flat, smooth and free of contamination. This technique, thanks to its high sensitivity and depth of analysis, has been used to identify the chemistry of the fibre-cement interface by analysing the surface of the fibre pulled-out from the cementitious matrix.

3.7 Shrinkage

3.7.1 Introduction

As discussed previously, in addition to the ageing process of the cement matrix and of the fibre-cement interface, drying shrinkage is of critical importance when attempting to predict the properties of a material with an expected design life of more than 100 years. Autogenous shrinkage also remains of importance in compositions with a low water/cement ratio. Composition B was tested to evaluate the effect of the environment on shrinkage: specimens were initially and then successively placed in different environments (air and water). Compositions A and C (as presented in section 3.2.3) were used to evaluate the effect of microsilica on drying shrinkage (specimen placed in air in laboratory). An experimental design was used to determine the effect of factors such as additive content, order of addition and mixing time on drying shrinkage and workability is measured using BS EN 1015: 1999.

3.7.2 Shrinkage measurement

Shrinkage was evaluated using beam specimens (500 mm x 100 mm x 100 mm; manufactured following BS EN 12390 Parts 1 and 2) with appropriate inserts to allow shrinkage to be determined along the 500 mm length (Morgan Sindall, 2011) as indicated in Figure 3-14a. The inserts embedded in the specimen were used as reference points to measure the change in length. Shrinkage beams were manufactured by pouring mixes into a mould (Figure 3-14b) and demoulding after 23 hours. The specimens were placed in a water tank at a controlled temperature of 20 °C to allow the specimen to achieve a standard temperature before measurement. The first measurement was taken an hour later i.e. 24 hours after the mix was poured into the mould. Shrinkage is measured by recording the change in length using a measurement cradle and reference bar (Figure 3-14b); the cradle has a centring device allowing the sample to be referenced in the same position each time a reading is taken. The
ambient temperature of the location where the specimen is placed, is also recorded along with the change of length to allow a correction of the length value measured (see below).

After the first measurement at 24 hours, the specimen is either placed in air to measure drying shrinkage or in water to measure autogenous shrinkage. As noted in the literature review, autogenous shrinkage is defined as the change of volume without any moisture transferred to the environment. Autogenous shrinkage can exist at the same time as drying shrinkage when the specimen is placed in air; however the value of the associated strain is lower for autogenous shrinkage than for drying shrinkage.

The measurements were recorded to the nearest 1 µm along with the age of the specimen. The values of change in length were plotted against time and given that such specimens are rarely kept at a constant temperature, a correction is usually required. The shrinkage, S, at a given point in time is thus given by:

\[
S = \left[ \frac{l_{t=0} - l_t}{l_{t=0}} \right] + (20 - T_t) \alpha_s
\]  

(3.10)

where \(S\) is the shrinkage in microstrain (µɛ), \(l_t\) is the length at a time \(t\), \(l_{t=0}\) is the initial length and \(T_t\) is the temperature at which the measurement at time \(t\) is taken and the coefficient of thermal expansion is taken as \(\alpha_s = 10 \times 10^{-5} \text{ m per } ^\circ\text{C}\). A temperature of 20 °C is taken as the reference.
3.7.3 Experimental Design method

As detailed in section 2.7.3, powder micro-silica can be used to control shrinkage, but does have some drawbacks associated with its use – in particular the workability of the fresh mixture can be much reduced. There are several factors that need to be considered including the dispersion of the micro-silica (especially given that the micro-silica is reputed to act as a pore-refiner) and its impact on the water content of the mix (the micro-silica has a large, active surface area and will tend to react with water from the mix. As noted in the literature review, particles of micro-silica tend to agglomerate, hence the mixing procedure, including the mixing time has its importance.

The study of several factors on a response would require many experiments which are time consuming and also costly. Plackett and Burman (1946) developed an experimental methodology enabling the estimation of the effect of several factors with two modalities each on a specific response, using a reduced number of experiments. This method is also called Screening Design and enables the selection of a small number of experiments to estimate the effect of a large number of factors on a response. The experimental design method was used to estimate the effect of three factors on shrinkage and workability, i.e.:

- Micro-silica (MS) content: 2.5 wt % and 5 wt %
- Mixing time: 5 mins and 10 mins
- The order of introduction of micro-silica: before water addition and after water
In order to determine the effect of these three factors on the responses of interest, four mixtures were prepared following the method suggested by Plackett and Burman (1946) and as presented in Table 3-4. The values measured were reported (A, B, C and D) in Table 3-4.

The orthogonal properties of these experiments enabled the use of a specific grid to estimate the mean effect of each factor on shrinkage and workability (Table 3-4). As an example, letters (A, B, C and D) indicated in Table 3-4 correspond to a response such as shrinkage or workability measurements, from which the values of the effect (K, L and M) is then calculated as detailed.

<table>
<thead>
<tr>
<th>Mixes</th>
<th>Micro-silica content</th>
<th>Introduction of MS</th>
<th>Mixing time</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.5 %</td>
<td>5 %</td>
<td>C+W+MS</td>
</tr>
<tr>
<td>1</td>
<td>/</td>
<td>A</td>
<td>A</td>
</tr>
<tr>
<td>2</td>
<td>/</td>
<td>B</td>
<td>/</td>
</tr>
<tr>
<td>3</td>
<td>C</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>4</td>
<td>D</td>
<td>/</td>
<td>D</td>
</tr>
<tr>
<td>Total</td>
<td>C+D</td>
<td>A+B</td>
<td>A+D</td>
</tr>
<tr>
<td>Number</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Average</td>
<td>(C+D)/2 = E</td>
<td>(A+B)/2 = F</td>
<td>(A+D)/2 = G</td>
</tr>
<tr>
<td>Effect Y-axis</td>
<td>(F - E) = K</td>
<td>(H - G) = L</td>
<td>(J - I) = M</td>
</tr>
<tr>
<td>X-axis</td>
<td>0.5</td>
<td>1.5</td>
<td>2.5</td>
</tr>
</tbody>
</table>

The standard deviation used for shrinkage and workability measurements is based on the average shrinkage values obtained with three specimens containing a similar percentage of micro-silica.

The experimental design method used does have limitations, in that, additional information such as possible interactions between the parameters evaluated, can be over-looked and more data would confirm the findings in order to build more effective models.

### 3.8 Summary

In this chapter, drawing on the information gathered from the literature review, and in the
context of the aims and objectives presented in section 1.3, the materials and methodologies used to characterise the mechanical performance, durability and shrinkage of ECCs were presented.

In particular, it should be noted that the mechanical testing of the tensile specimens has required significant deviation from the “Japanese standard”. This may simply be due to the effect of increasing the thickness of the specimens tested, but it is an effect that must be borne in mind when considering the results of the current work and that of other researchers.

In the following chapters, the results from the testing programme are presented and discussed: Chapter Four focusses on the mechanical properties of ECCs; Chapter Five looks at the durability and Chapter Six presents data on shrinkage.
4 Mechanical performance

4.1 Introduction
In the previous chapter, the materials and methods used in the current study were presented: these have been chosen with reference to the literature. In this chapter, the results from the programme of mechanical testing are presented and discussed. First, the tensile performance of ECCs at 27 days is presented along with a comparison with unreinforced cements. Then, the effects of process, fibre type, casting method and specimen thickness are detailed along with a fractographic analysis of the fractured surfaces of the specimens mechanically tested. Fibre dispersion and orientation data are also presented for a number of samples. Results from single fibre pull-out testing of fibres embedded in cement at 28 days are shown: these data enable interfacial property data to be determined, which are related to the tensile performance of the material. The stress-strain data from the tensile tests are considered in the light of the theoretical models outlined in the literature review (section 2.4) which require fibre orientation data and interface properties as input parameters. ECC specimens have also been tested in flexure; the effects of process and fibre type are evaluated and the results are presented here. Finally, physical properties such as density and porosity are determined and linked to the tensile and flexure test parameters in order to make a correlation with the microstructure of the ECC which is influenced by the process and composition. The correlation between these properties may indicate lines of enquiry for further investigation and also provide guidance for specific protocols to be employed on site in future civil engineering applications. Hence, valuable information could be achieved enabling a better control of the mechanical properties of the ECC for its application as a secondary lining in tunnels.

4.2 Tensile testing

4.2.1 Introduction
Traditionally cementitious materials are expected to perform better in compression than in tension. Whilst there are steps that can be taken to improve the tensile performance, the tensile properties will always be of concern for a structure that may be subjected to a load in pure tension or flexure. Tensile testing therefore represents the sternest challenge to any cementitious material. To study the response of the ECC in tension, dog-bone specimens have been manufactured. Specimens were made with different fibre and process types (see Table
4-1); three specimens were manufactured for each fibre and process type and three specimens without fibres were also cast as a control. The tensile properties of the specimens made with different fibre and process type were determined and compared. The effect of process and fibre type, and casting method on the mechanical performance is detailed. The effect of the specimen thickness is compared with the literature data from previous studies. Fractographic examination of the fracture surfaces enable the extent of the fibre pull-out, which is linked to the mechanical performance of the ECC, to be assessed. This is key to the tensile performance of the ECC.

Table 4-1: Tensile testing experimental programme (composition A)

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Description (Fibre type/Process)</th>
<th>Workability (mm)</th>
<th>Age when tested (days)</th>
<th>Casting method</th>
</tr>
</thead>
<tbody>
<tr>
<td>C6256</td>
<td>T2/P1</td>
<td>175</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6257</td>
<td>T2/P1</td>
<td>175</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6258</td>
<td>T2/P1</td>
<td>175</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6522</td>
<td>T1/P2</td>
<td>208.5</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6523</td>
<td>T1/P2</td>
<td>208.5</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6524</td>
<td>T1/P2</td>
<td>208.5</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6541</td>
<td>T2/P1</td>
<td>175</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6542</td>
<td>T2/P1</td>
<td>175</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6543</td>
<td>T2/P1</td>
<td>175</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C6544A</td>
<td>T1/P1</td>
<td>175</td>
<td>27</td>
<td>slab</td>
</tr>
<tr>
<td>C6544B</td>
<td>T1/P1</td>
<td>175</td>
<td>27</td>
<td>slab</td>
</tr>
<tr>
<td>C6544C</td>
<td>T1/P1</td>
<td>175</td>
<td>27</td>
<td>slab</td>
</tr>
<tr>
<td>C7097</td>
<td>without fibres</td>
<td>/</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C7098</td>
<td>without fibres</td>
<td>/</td>
<td>27</td>
<td>mould</td>
</tr>
<tr>
<td>C7099</td>
<td>without fibres</td>
<td>/</td>
<td>27</td>
<td>mould</td>
</tr>
</tbody>
</table>

4.2.2 Comparison of unreinforced and reinforced cement

4.2.2.1 Introduction
The methodology for tensile testing was outlined in section 3.4.2. The effect of adding the fibres to each specific composition of the cementitious matrix was investigated according to Table 4-1. The appearance of the damaged dog-bone specimens with and without fibres tested in tension are first compared (section 4.2.2.2) and then the stress-strain curves obtained from these tests are described (section 4.2.2.3), followed by the presentation of similar data for the rest of the test programme (section 4.2.3).

4.2.2.2 Single and Multiple cracking
Figure 4-1 compares the appearance of the dog-bone specimens without fibres (C7097, Figure
4-1.a) and with fibres (C6523, Figure 4-1.b) tested in tension. As might be expected, the specimen without fibres presents only one crack immediately followed by the failure of the specimen, thus demonstrating brittle behaviour, whereas the specimen containing microfibres exhibits the multiple cracking behaviour before failure as described in the literature (section 2.5.2).

![Single crack](image1)

![Multiple fine cracks](image2)

**Figure 4-1**: Pictures taken after tensile testing of (a) a thin dog-bone specimen without fibres (C7099) and (b) a thin dog-bone specimen with fibres (C6523) according to the ECC design mixture

### 4.2.2.3 Pseudo-ductile behaviour of ECCs

Figure 4-2 enables the stress-strain response of the cementitious matrix with and without added fibres to be compared. The specimen without fibres fails in a brittle manner: the tensile stress increases linearly with the strain until sudden failure (specimen C7099). Specimen C6523 containing fibres, presents a typical response of an ECC subjected to a tensile stress. The response is characterised by three stages:

1. **Pre-cracking zone**: a linear-elastic response up to the formation of the first crack in the matrix.
2. **Multiple-cracking zone**: a pseudo-plastic region characterised by the formation of multiple fine cracks. The matrix stress at the crack planes is zero but away from the crack, the load is transferred from the fibre to the matrix via the fibre-matrix interface leading to an increase of matrix stress and the possibility of further matrix cracks within the gauge length of the specimen.
iii. Post-cracking zone: this starts when a major crack (wider than the previous cracks) starts to form, the material is held together by the bridging fibres only up to the complete failure of the specimen.

![Typical stress-strain curves for specimens tested in tension](image)

**Figure 4-2: Typical stress-strain curves for specimens tested in tension: a specimen without fibres (C7099) and a specimen containing 2 vol. % fibres (C6523)**

These tests confirmed the hypothesis made in the literature review: the incorporation of polymeric micro-fibres would lead to pseudo-ductility of the ECCs associated with the formation of multiple cracks under applied stress.

As predicted in section 3.4.2.2, the results associated with the new geometry revealed an improved performance (a strain of more than 2 %) associated with multiple fine cracks within the gauge length of the specimen compared with the specimens made using the initial geometry with sharp edges and an associated stress concentration (section 3.4.2.3). This confirmed the hypothesis that such a geometry without sharp edges would promote enhanced mechanical performance. However, as detailed in section 2.5.2, a tensile strain of 5.3 % is found in the literature (Li, 2003), hence the objective of this chapter is also to have a better understanding of the difference and variability observed.

### 4.2.3 Effect of process and fibre type on mechanical performance

Having modified the test geometry, further tensile testing was carried out in order to evaluate
the effect of process and fibre type on mechanical behaviour, in accordance with the test matrix in Table 4-1. Three specimens were manufactured per fibre and process type; specimens were tested at 27 days.

The results are shown in Figures 4-3 and 4-4. All the specimens tested show multiple-cracking and hence a pseudo-ductile behaviour under tensile stress. There is variation between similar specimens, but this is not unexpected and is typical behaviour of (cured) cementitious material. It is highly likely that variability is in part a consequence of the nature of the cementitious matrix, which consists partly of crystals, and the variability in the microstructure linked with the manufacturing process, as well as the presence of porosity. The non-uniformity of the fibre distribution may also have a role in the sample-to-sample variation. Figure 4-5 provides another illustration of the variability in the capacity of dog-bone samples of ECC tested in tension to undergo controlled multiple cracking. The arrows indicate clusters of cracks. As expected, the specimen that shows most cracking prior to failure also shows the greatest strain-to-failure $\varepsilon$ (here taken from the point at which the first crack occurs), whereas the smallest number of cracks corresponds with the lowest strain-to-failure. Comparison of the samples with the corresponding load-displacement (stress-strain) responses, see Figure 4-3, indicates that the formation of these clusters of cracks correlate with one or more load-drop events. Further analyses are required to explain the variability associated with the results observed, including the analysis of the physical properties such as density and porosity; some of these are presented later in this chapter.
Comparing the results in detail, Figure 4-3 shows that when Process P1 was used, specimens manufactured using fibre T2 exhibit a higher maximum stress ($\sigma_{\text{max}}$) whereas specimens using fibre T1 show a higher ultimate strain. This is likely to be a result of a different fibre-matrix interface in the two systems and/or a different fibre distribution and orientation caused by
the rigidity of the resin-bundling, which is consistent with the literature. Possibly fibre T2 promotes enhanced matrix strength due to the resin-bundling, whereas for fibre T1 specimens, first cracking at low stresses enables a higher strain after first crack. These results suggest that fibre T1 would have an advantage over fibre T2 when using Process P1, hence promoting a larger strain to failure.

Turning to Figure 4-4, specimens containing fibre T1 and manufactured with Process P2 present the highest maximum stresses (up to 5 MPa) and comparable ultimate strains when compared to those manufactured with Process P1. It seems that fibre T1, as in Figure 4-3, promotes a higher strain and the use of Process P2 enables a higher maximum stress. Process P2 enables, perhaps, a better fibre dispersion/orientation and therefore better mechanical performance. Based on the results from these experiments, performance is optimised using fibre T1 with Process P2. A value of workability was measured for each batch manufactured. As detailed in Chapter 2, an acceptable workability is very relevant when applying ECCs on-site. Table 4-1 shows that using fibre T1 with P2 gives the highest workability with a value of 208.5 mm. Further analyses are required to understand the differences in mechanical performance observed in more detail; this will include consideration of fibre orientation effects.
Figure 4-5: Photographs of dog-bone ECC specimens made from the same batch of material following testing (a) sample reference C6541, $\varepsilon = 1.04$ (b) C6542, $\varepsilon = 0.33$ and (c) C6543, $\varepsilon = 2.24$; the quantity $\varepsilon$ represents the strain to failure after the first crack (load-drop) occurs. The arrows indicate clusters of cracks, each of which correlates with one or more load-drop events on the load-displacement (stress-strain) response, see Figure 4-3.

### 4.2.4 Effect of the casting method – Moulded and machined specimens

The influence of the casting method (either moulded or machined specimen) on the mechanical performance was considered: the normal process saw the mix cast in dog-bone shaped moulds, but for comparison, slabs (section 3.3.2) were also cast and dog-bone samples machined via an abrasive water jet cutting process. These machined specimens were tested in tension in the usual way.

Figure 4-6 compares the mechanical performance of specimens cast using the modified dog-bone mould and specimens of the same geometry machined from a slab. The moulded
specimens exhibit both superior strength and strain to failure compared with those cut out. A relatively low maximum stress (up to 2.5 MPa) and strain after the first crack (up to 0.25 %) are presented by the specimens cut from the slab compared with the ones made in a mould (maximum stress up to 4.3 MPa and strain after first crack up to 2.24 %). This suggests that the casting method could influence the physical properties, as well as the fibre dispersion and orientation, or possibly the cutting process could promote the presence of micro-cracks at the edge of the specimen cut from the slab, although care has been taken to ensure the good choice of cutting parameters and there was no evidence that the cutting process damaged the specimen (section 3.3.2). It seems reasonable to suggest that the use of the mould, due to its smaller size compared with the slab, could align the fibres in the loading direction and hence promote tensile performance. In contrast, the specimen cut from the slab leads to a more random distribution of fibres and where fibres present at the edges are cut, these fibres would be expected to contribute less to the mechanical performance. This is discussed further in section 4.3.

![Figure 4-6: Stress-strain results for specimens tested in tension: comparison of specimens cast in moulds with specimens cast in a slab](image)

**4.2.5 Effect of specimen thickness**

As noted in the introduction, of specific interest in the present work was the effect of specimen thickness. Figure 4-7 compares the mechanical performance as obtained in this study (using a specimen thickness of 30 mm) with data obtained in previous studies using
different thickness section specimens and using fibres of a similar length and composition (Li and Weiman, 2003; Kanakubo, 2006; Kim et al., 2007). It appears that the ultimate strain decreases to reach a plateau whereas the maximum stress slightly increases or remains constant with increasing specimen thickness.

As detailed in the literature (section 2.4.4), fibre orientation might be expected to play a major role in mechanical performance. Therefore, a higher maximum stress might be expected with thinner specimens where the fibres are more likely to align in the tensile loading direction (section 2.4.4). Instead, Figure 4-7 shows that the specimen thickness impacts primarily on the maximum tensile strain, i.e. the extent of pseudo-ductility following first cracking, which seems to decrease with specimen thickness. This is not surprising as the fibres are transferring the load to the matrix for the formation of further cracks. The trend of decreasing maximum strain with increasing thickness is most likely a function of increasing variability in the fibre orientation and distribution. It is reasonable to suppose that using thin sections of up to 13 mm with fibres of length of about 10 mm, is more likely to lead to the manufacture of specimens with most fibres orientated in the tensile axis direction. This orientation effect might be expected to lead to an increased strength, but if strength is controlled by fibre debonding and pull-out (and this is not sensitive to orientation), then maybe strength staying fairly constant is not surprising. The corresponding failure mechanisms will be considered further in the section concerned with strength modelling.

The data in Figure 4-7 suggest that for this specific ECC composition, a minimum useful strain-to-failure of 4 % would be achieved at a maximum section thickness of 20 mm. It may be that thicker sections might require a higher fibre volume fraction ($V_f$) to work effectively and enhance pseudo-ductility or a better control of the manufacturing process, leading to greater alignment of the fibres in preferred orientations, although there may be a subsequent impact on the workability. For hydraulic pressure tunnels, the tensile strain can reach 0.5 %. For ECC design however, where there will be reliance on multiple-cracking, 1 % could be a minimum requirement to provide ductility equivalent to reinforced concrete. The data in Figure 4-7 suggest that a strain of 1 % can be achieved over the full range of thicknesses shown.
4.2.6 Fractography

Fractured surfaces from the tensile samples were examined, as discussed in section 3.5.3.2. In this way, the matrix porosity could be assessed and observations regarding the fibre distribution and the fibre-matrix interface could be made. The fibres can be identified by their size and shape. Small samples were cut from the cracked faces of the tensile specimens and gold coated (four coatings of 99.9% gold, each 2 nm thick) to make them conductive for SEM examination.

In the current study, the fracture surface of an ECC tested in tension (specimen C6543, a sample which performed relatively well in tension) was examined using SEM, see Figure 4-8. An overall perspective is given in Figure 4-8a which shows a cluster of fibres from the fracture surface, whilst Figures 4-8b-d present examples of particular behaviour. Figure 4-8b corresponds to an entire (intact) fibre with some PVA material detached from its surface; this type of fibre could contribute well to the pseudo-ductility of the ECC composite. Figure 4-8c provides evidence suggesting that the fibre-matrix bond-strength is, in places, greater than the strength of the cementitious matrix, leading to a cohesive failure of the matrix. This type
of behaviour may contribute in a limited way to the pseudo-ductility of the composite, as shown in the literature (Redon et al., 2001). Finally, Figure 4-8d shows a damaged fibre in which the fibre end appears narrower compared with its original diameter; this type of behaviour will make only a small contribution to the pseudo-ductility of the ECC in tension and is usually seen at the end of testing. Boshoff et al. (2009) associated the fibre appearance at the end of a single test with the interfacial shear resistance against the displacement, and suggested that a fibre that ruptures during single fibre pull-out testing such as the one shown in Figure 4-8d, is associated with a sudden drop of force whereas a fibre that pulls-out exhibits a steady load reduction until a zero load resistance is achieved. This idea is explored further in section 5.3.4 dealing with the single fibre pull-out test at different ages.

![Figure 4-8: Secondary scanning electron photomicrographs (SEM) from a fractured ECC specimen (a) low magnification image showing a number of pulled-out fibres, (b)-(d) higher magnification of single fibres showing different interface and fracture behaviour (20 kV) – Specimen C6543](image)

The images confirm the ability of the fibres, as detailed in the literature review, to pull-out from a cementitious matrix under stress instead of breaking. This behaviour is in line with the enhanced mechanical performance measured for the ECC compared to the unreinforced
matrix, leading to the observed pseudo-ductile behaviour under stress.

4.3 Characterisation of fibre dispersion and orientation

4.3.1 Fibre dispersion and distribution

4.3.1.1 Introduction
As mentioned in section 2.5.3.4, a good fibre dispersion in ECCs is crucial for the achievement of the optimum mechanical performance. The fibre dispersion is evaluated using images obtained by SEM operating in backscattered electron mode; photomicrographs of sections from the specimen cured in air were taken at different locations in the specimen and at different magnifications.

4.3.1.2 Low magnification dispersion
Figure 4-9 shows the fibre dispersion in an ECC specimen at low magnification. A reasonably uniform fibre dispersion is visible for a low fibre volume fraction ($V_f = 2$ vol.%) with almost no defects and where fibre clumping does not seem to be an issue. This confirms the suitability of Process P1 promoting an excellent fibre dispersion (with fibre type T2). In this case, the fibre density is evaluated as 16.4 fibres/mm$^2$ occupying 2.05 % of the total surface area analysed, which is consistent with the introduction of fibres in the mixture at 2 vol. %.

Furthermore, most fibres exhibit a reasonably circular shape, where the fibres are cut perpendicular to the surface analysed, with only a few fibres showing an elliptical shape. This could be explained by the existence of a preferential fibre orientation promoted by processing and also the casting method (the specimen being cast in a mould). This needs to be taken into consideration when analysing the mechanical performance of the material.
4.3.1.3 Intermediate magnification dispersion

Figure 4-10 shows the fibre dispersion at an intermediate magnification. The fibre dispersion seems excellent with the fibres equally dispersed between the sand and cement grains, confirming the observation made at larger scale. The fibre density is estimated at 10.7 fibres/mm$^2$, this value being smaller than the one obtained for a low magnification (16.4 fibres/mm$^2$). This is perhaps not surprising as the smaller the area of the sample, the greater the expected spread in the measured fibre area density.
Figure 4-10: Backscattered electron image (SEM) of a typical ECC specimen showing the fibre dispersion and distribution at an intermediate magnification

4.3.1.4 High magnification dispersion

Figure 4-11 shows the fibre dispersion at high magnification. Again the fibre dispersion between the sand and cement is reasonably good; the space between the fibres seems roughly uniform, confirming the observations made at lower magnifications. The higher magnification image suggests that there is no macroscopic microstructural irregularity (such as a defect or air bubble) at the fibre-matrix interface. The fibre density is estimated at 29.4 fibres/mm². This value is slightly higher than the ones obtained at lower magnifications, which is consistent with the suggestion that at higher magnification, there is inevitably a departure from the average volume fraction.
4.3.2 Fibre orientation

Fibres can be classified according to their orientation (Lee et al., 2009). In the current work, approximately 300 images were taken of polished surfaces from three specimens: C6523, C6543 and C6544C. The fibres visible in the images were then counted and classified depending on their orientation using ImageJ (open source digital image analysis software, ImageJ 1.46/Java 1.6.0_20 (64-bit)). Figure 4-12 illustrates an example of a backscattered electron image; a circular fibre shape is aligned with the tensile testing axis (0 to 20 °) whereas an elliptical shape suggests a certain orientation of the fibre (above 20 °) – the greater the elongation of the ellipse, the larger the angle of inclination of the fibre to the specimen loading direction.
Figure 4-12: Backscattered electron image (SEM) of a polished surface from an ECC sample (C6544C) showing typical porosity and a range of fibre sections corresponding to different orientations.

Figure 4-13a compares the quantity of fibres for each orientation for tensile specimens cast in a mould (C6523 and C6543) and a specimen cut from a slab cast in a similar way (C6544C). These results suggest a greater proportion of fibres aligned with the tensile axis (0 - 30°) for a specimen cast in a mould, especially C6523, whereas the specimen cast in a slab (C6544C) seems to exhibit a more random orientation of the fibres. Specimen C6543 was also cast in the mould but using the lower workability process P1 and this may be the reason for less fibre alignment. It is also possible that the casting method introduced a random distribution of the fibres or a degree of alignment and the low workability of the mix leads to this alignment remaining in the specimen cut subsequently from the slab resulting in a slight fibre alignment in the opposite direction to the tensile load.
As might be expected, and as can be seen in Figure 4-13b, the sample with the greatest maximum stress and strain after first crack is the one with the highest proportion of fibres
aligned in the direction of the applied stress. Indeed, specimen C6523, exhibiting enhanced performance, shows more than 50% of its fibres aligned along the tensile testing axis (0 - 30°) compared with specimen C6544C which has a more random orientation of fibres and lower mechanical performance. This may suggest that Process P2 (enabling a greater workability of the ECC mixture and where the fibres are put last in the mixture) would enable a better flexibility in the orientation of the fibres, which may lead to fibre alignment along the tensile testing axis when casting in a mould.

Data on fibre orientations (Figure 4-13a) are used to calculate $\eta_\theta$ for each specimen. Table 4-2 presents values of $\eta_l$ and $\eta_\theta$ (see section 2.4.4) for each specimen tested, based on equations 4.1, 4.2 and 4.3:

$$\eta_l = 1 - \frac{\tanh \left( \frac{\beta L}{2} \right)}{\left( \frac{\beta L}{2} \right)}$$ \hspace{1cm} (4.1)

$$\beta = \frac{2\pi G_m}{E_f A_f \ln \left( \frac{R}{R_f} \right)}$$ \hspace{1cm} (4.2)

$$\eta_\theta = \sum_{i=0}^{\theta_{max}} V_{f_i} \cos^4 \theta_i$$ \hspace{1cm} (4.3)

The greater orientation in specimen C6523, which is apparent in Figure 4-13, leads to a value for $\eta_\theta$ of 0.51, while the values for the other two specimens, C6543 and C6544C (0.37 and 0.31 respectively) are very close to that for a material with quasi-isotropic properties. The improved mechanical performance of C6523 is in line with this greater degree of orientation (Table 4-2).
Table 4-2: Fibre orientation data for ECC samples based on T1 fibres taken from moulds (prepared by two different manufacturing routes: specimens C6543 and C6523) and a slab (specimen: C6544C) (SI)

<table>
<thead>
<tr>
<th>Angle</th>
<th>θi</th>
<th>%</th>
<th>Vfi</th>
<th>ηθi</th>
<th>Nbr (%)</th>
<th>Vfi</th>
<th>ηθi</th>
<th>Nbr (%)</th>
<th>Vfi</th>
<th>ηθi</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 - 20 °</td>
<td>10</td>
<td>0.17</td>
<td>2073</td>
<td>15.95</td>
<td>0.16</td>
<td>0.150</td>
<td>812</td>
<td>6.88</td>
<td>0.07</td>
<td>0.065</td>
</tr>
<tr>
<td>20 - 30 °</td>
<td>25</td>
<td>0.44</td>
<td>4216</td>
<td>32.44</td>
<td>0.32</td>
<td>0.219</td>
<td>2380</td>
<td>20.17</td>
<td>0.2</td>
<td>0.136</td>
</tr>
<tr>
<td>30 - 40 °</td>
<td>35</td>
<td>0.61</td>
<td>2864</td>
<td>22.03</td>
<td>0.22</td>
<td>0.099</td>
<td>2755</td>
<td>23.35</td>
<td>0.23</td>
<td>0.105</td>
</tr>
<tr>
<td>40 - 50 °</td>
<td>45</td>
<td>0.79</td>
<td>1410</td>
<td>10.85</td>
<td>0.11</td>
<td>0.027</td>
<td>2168</td>
<td>18.37</td>
<td>0.18</td>
<td>0.046</td>
</tr>
<tr>
<td>50 - 60 °</td>
<td>55</td>
<td>0.96</td>
<td>925</td>
<td>7.12</td>
<td>0.07</td>
<td>0.008</td>
<td>1662</td>
<td>14.08</td>
<td>0.14</td>
<td>0.015</td>
</tr>
<tr>
<td>60 - 70 °</td>
<td>65</td>
<td>1.13</td>
<td>1076</td>
<td>8.28</td>
<td>0.08</td>
<td>0.003</td>
<td>1367</td>
<td>11.58</td>
<td>0.12</td>
<td>0.004</td>
</tr>
<tr>
<td>70 - 90 °</td>
<td>80</td>
<td>1.4</td>
<td>434</td>
<td>3.34</td>
<td>0.03</td>
<td>3.00E-05</td>
<td>656</td>
<td>5.56</td>
<td>0.06</td>
<td>5.00E-05</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td></td>
<td>12998</td>
<td></td>
<td></td>
<td>100</td>
<td></td>
<td></td>
<td>1</td>
<td>0.506</td>
</tr>
</tbody>
</table>

4.4 Single fibre Pull-Out testing

4.4.1 Load-displacement data

Single fibre pull-out tests of a fibre from a cementitious matrix have been carried out on samples aged 28 days so as to characterise the fibre-cement interface and to correlate the data obtained with the mechanical performance measured during tensile testing. As indicated in section 3.4.4, these tests were carried out at the Leibniz Institute of Polymer Research (Leibniz-Institute für Polymerforschung) in Dresden, Germany and the test data were provided for analysis. The procedure for preparing the samples is also detailed in section 3.4.4., which starts with the preparation of the matrix and then the insertion of the single fibre.

A total of 21 tests were carried out. These gave a range of stress-strain responses, and three stages were identified (Figure 4-14):

- An elastic region (up to initial de-bonding of the fibre) during which the load increases up to a specific value \( P_a \); by the end of this stage the embedded fibre end has not moved; the elastic region corresponds to the stretching of the fibre.
- At the end of the elastic region, the load drops suddenly from \( P_a \) to \( P_b \); this is particularly the case for PVA fibres, where this sudden load drop corresponds to the chemical bond at the fibre-cement interface being broken (Redon et al., 2001). In the literature, it is assumed that this sudden failure means that debonding at the interface
is governed by a fracture mechanism-based criterion rather than by a strength-based criterion (Leung and Li, 1990; Li and Stang, 1997).

- Once the fibre has debonded, there is a slippage regime during which the fibre end is now moving and the fibre sliding is resisted by frictional forces at the fibre-matrix interface.

In the current study, three different cases have been identified for the final regime (fibre slippage) as illustrated in three tests on samples from the batch aged for 28 days (Figure 4-14):

- A constant friction mechanism characterised by a value of the slip-hardening coefficient $\beta = 0$ (Figure 4-14a).
- A slip-hardening mechanism leading to a linear increase of the load with the displacement and the load increases as a result of the frictional forces present at the fibre-cement interface. A value for $\beta$ of greater than zero characterises this type of behaviour (Lin and Li, 1997), see Figure 4-14b.
- A slip-softening mechanism, the load decreases rapidly during the pull-out regime. A value for $\beta$ of less than zero characterises this type of behaviour (Lin and Li, 1997), see Figure 4-14c.

According to the literature (Redon et al., 2001), one is more likely to obtain slip-hardening behaviour with polymeric fibres than other fibre types (ceramic or metal for example) as polymeric fibres are less hard than the surrounding matrix and so can damage easily within the matrix, causing a jamming effect. It is interesting to note that the embedded lengths are very similar for the three samples for which data are shown in Figure 4-14, suggesting that there is no particular effect of embedded length on the friction mechanisms seen, which are different.
Figure 4-14: Single fibre pull-out curve for (a) constant friction $\beta = 0$, (b) slip-hardening $\beta > 0$ and (c) slip-softening $\beta < 0$
### 4.4.2 Interfacial property data

From the single fibre pull-out tests at 28 days, three interfacial parameters are determined:

- $\tau_d$: interfacial shear strength, characterising the initial debonding mechanism of the fibre from the cementitious matrix (Zhandarov and Mäder, 2005):

$$
\tau_d = \frac{P_a}{\pi d_f l_e}
$$

(4.4)

- $G_d$: chemical debonding energy, which is derived from the second stage of the pull-out testing curve, as it is closely linked to the sudden load drop once the fibre/cement chemical bond is broken (Redon et al., 2001):

$$
G_d = \frac{2(P_a - P_b)^2}{\pi^2 E_f d_f^3}
$$

(4.5)

- $\tau_0$: frictional bond strength (Redon et al., 2001), characterises the last stage of the single fibre pull-out testing curve:

$$
\tau_0 = \frac{P_b}{\pi d_f l_e}
$$

(4.6)

- $\beta$: parameter characterising the slippage regime

The values for each parameter were calculated using the fibre diameter $d_f$ and the exact fibre embedded length $l_e$ for each sample. All values are shown in Table 4-3 together with an average value and a standard deviation for each parameter.

The mean value of $\tau_d$ is 3.55 MPa with a standard deviation of 1.70 MPa. Some specimens gave results which are outliers and possibly erroneous. However, a value for $\tau_d$ of 3.33 MPa is estimated when discarding the highest and lowest values, and $\tau_d$ equals 3.30 MPa when discarding the two lowest and highest values. Discarding the highest and lowest values do not appear to distort the mean value significantly.
Table 4-3: Interfacial bond parameters

<table>
<thead>
<tr>
<th>Test No</th>
<th>(d_f) (µm)</th>
<th>(l_e) (µm)</th>
<th>(P_a) (N)</th>
<th>(P_b) (N)</th>
<th>(\tau_d) (MPa)</th>
<th>(G_d) (J.m(^{-2}))</th>
<th>(\tau_0) (MPa)</th>
<th>Slippage regime type</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>39.39</td>
<td>970.06</td>
<td>0.34</td>
<td>0.18</td>
<td>2.83</td>
<td>2.12</td>
<td>1.50</td>
<td>(\beta = 0)</td>
</tr>
<tr>
<td>2</td>
<td>40.96</td>
<td>931.25</td>
<td>0.22</td>
<td>0.09</td>
<td>1.79</td>
<td>1.15</td>
<td>0.75</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>3</td>
<td>43.28</td>
<td>527.83</td>
<td>0.37</td>
<td>0.23</td>
<td>5.10</td>
<td>1.12</td>
<td>3.23</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>4</td>
<td>42.18</td>
<td>339.30</td>
<td>0.43</td>
<td>0.19</td>
<td>9.50</td>
<td>3.66</td>
<td>4.31</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>5</td>
<td>38.04</td>
<td>965.79</td>
<td>0.31</td>
<td>0.18</td>
<td>2.70</td>
<td>1.73</td>
<td>1.52</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>6</td>
<td>38.57</td>
<td>1073.87</td>
<td>0.36</td>
<td>0.18</td>
<td>2.77</td>
<td>3.05</td>
<td>1.34</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>7</td>
<td>38.81</td>
<td>977.42</td>
<td>0.41</td>
<td>0.13</td>
<td>3.40</td>
<td>6.46</td>
<td>1.11</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>8</td>
<td>40.37</td>
<td>466.77</td>
<td>0.31</td>
<td>0.15</td>
<td>5.24</td>
<td>1.95</td>
<td>2.55</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>9</td>
<td>40.31</td>
<td>1044.92</td>
<td>0.31</td>
<td>0.12</td>
<td>2.34</td>
<td>2.85</td>
<td>0.89</td>
<td>(\beta = 0)</td>
</tr>
<tr>
<td>10</td>
<td>39.65</td>
<td>974.74</td>
<td>0.34</td>
<td>0.10</td>
<td>2.77</td>
<td>4.56</td>
<td>0.82</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>11</td>
<td>29.82</td>
<td>497.02</td>
<td>0.24</td>
<td>0.16</td>
<td>5.24</td>
<td>1.25</td>
<td>3.50</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>12</td>
<td>40.78</td>
<td>617.68</td>
<td>0.32</td>
<td>0.11</td>
<td>4.08</td>
<td>3.33</td>
<td>1.42</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>13</td>
<td>36.66</td>
<td>942.66</td>
<td>0.32</td>
<td>0.15</td>
<td>2.98</td>
<td>3.22</td>
<td>1.34</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>14</td>
<td>45.67</td>
<td>1005.02</td>
<td>0.33</td>
<td>0.11</td>
<td>2.27</td>
<td>2.48</td>
<td>0.78</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>15</td>
<td>36.52</td>
<td>982.20</td>
<td>0.32</td>
<td>0.13</td>
<td>2.85</td>
<td>3.64</td>
<td>1.19</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>16</td>
<td>39.10</td>
<td>721.93</td>
<td>0.27</td>
<td>0.15</td>
<td>3.00</td>
<td>1.22</td>
<td>1.65</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>17</td>
<td>41.57</td>
<td>826.28</td>
<td>0.40</td>
<td>0.17</td>
<td>3.74</td>
<td>3.99</td>
<td>1.54</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>18</td>
<td>37.76</td>
<td>1074.66</td>
<td>0.37</td>
<td>0.17</td>
<td>2.89</td>
<td>3.84</td>
<td>1.30</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>19</td>
<td>40.70</td>
<td>1014.69</td>
<td>0.36</td>
<td>0.13</td>
<td>2.77</td>
<td>4.08</td>
<td>0.97</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td>20</td>
<td>38.44</td>
<td>627.15</td>
<td>0.33</td>
<td>0.11</td>
<td>4.30</td>
<td>4.20</td>
<td>1.44</td>
<td>(\beta &gt; 0)</td>
</tr>
<tr>
<td>21</td>
<td>41.01</td>
<td>1593.09</td>
<td>0.41</td>
<td>0.19</td>
<td>2.02</td>
<td>3.62</td>
<td>0.94</td>
<td>(\beta &lt; 0)</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>39.50</strong></td>
<td><strong>865.44</strong></td>
<td><strong>0.34</strong></td>
<td><strong>0.15</strong></td>
<td><strong>3.55</strong></td>
<td><strong>3.03</strong></td>
<td><strong>1.62</strong></td>
<td><strong>28 days</strong></td>
</tr>
<tr>
<td><strong>Std dev.</strong></td>
<td><strong>3.09</strong></td>
<td><strong>281.59</strong></td>
<td><strong>0.05</strong></td>
<td><strong>0.04</strong></td>
<td><strong>1.70</strong></td>
<td><strong>1.36</strong></td>
<td><strong>0.96</strong></td>
<td></td>
</tr>
</tbody>
</table>

The value of \(G_d\) is equal to 3.03 J m\(^{-2}\) with a standard deviation of 1.36 J m\(^{-2}\). The values obtained are typical of an oiled fibre tailored for ECCs as detailed in the literature (Redon et al., 2001) where an average value of 2.8 J m\(^{-2}\) is given for \(G_d\). Redon et al (2001) emphasise the benefits of the oil coating reducing \(G_d\) so as to enhance the pseudo-ductile behaviour of the composite as well as lowering the slip-hardening effect to avoid fibre rupture. In order to achieve these goals, an oil coating is applied (Redon et al., 2001). A value for \(G_d\) of 2.95 J m\(^{-2}\) is estimated when discarding the highest and lowest values, whereas \(G_d\) equals 2.96 J m\(^{-2}\) when discarding the two lowest and highest values.

The mean value of \(\tau_0\) is 1.62 MPa with a standard deviation of 0.96 MPa; as expected, this value is within the range of the optimal frictional bond (\(\tau_0\) to be between 1-2 MPa) suggested for the composite to achieve pseudo-ductile behaviour under tensile stress as detailed in Chapter 2 (Li, 2002b). This also demonstrates that the surfaces of the fibres used were specifically tailored to the cementitious matrix to offer an appropriate resistance when the ECC is under stress enabling the pseudo-ductility of the composite. A value for \(\tau_0\) of 1.53 MPa
is estimated when discarding the highest and the lowest values, whereas $\tau_0$ equals 1.46 MPa when discarding the two lowest and highest values showing a low variability in the results.

### 4.5 Application of models for mechanical behaviour in tension

#### 4.5.1 Introduction

As detailed in section 2.4, theoretical models have been established for fibre reinforced materials and in the present section, relevant models are used to represent the tested ECC material. In this section and in the light of ECC behaviour, the ACK model up to and including first crack is applied here, then the critical volume fraction of fibres is discussed, and observations on the crack spacing are detailed, followed by recommendations for a model to predict the strength of ECCs, post first crack.

#### 4.5.2 ACK model

##### 4.5.2.1 Introduction

ACK theory (for aligned fibres) as discussed in section 2.4.5, defines three zones in the stress-strain behaviour of a brittle fibre reinforced composite matrix: the pre-cracking zone, the multiple cracking zone and the post-cracking zone. The mechanical testing results and fibre orientation data obtained for specimens C6523, C6543 and C6544C are applied to the ACK model to predict the material properties of ECC materials.

##### 4.5.2.2 Behaviour before first crack

The pre-cracking zone corresponds to the linear elastic region (section 2.4.5), thus enabling the calculation of the composite Young’s modulus $E_c$ based on equations 4.7 and 4.8.

$$ E_c = E_f V_f + E_m V_m $$

$$ V_m = 1 - V_f $$

Using $E_f = 40$ GPa, $V_f = 0.02$, $E_m = 13$ GPa (Neville, 2000) and $V_m = 0.98$ gives an $E_c$ value for ECC of 13.54 GPa.

However, equation 4.7 is based on continuous aligned fibres, whereas ECCs exhibit randomly distributed fibres, hence the need to introduce equations 4.9 and 4.10:
\[ E'_c = E_f V_f^* + E_m V_m \]  
(4.9)

\[ V_f^* = \eta \eta_\theta V_f \]  
(4.10)

where, based on elastic load transfer at the interface:

\[ \eta_\ell = 1 - \left[ \tanh \left( \frac{\beta L}{2} \right) \right] / \left[ \left( \frac{\beta L}{2} \right) \right] \]  
(4.11)

\[ \beta = \frac{2 \pi C_m}{E_f A_f \ln \left( \frac{R}{R_f} \right)} \]  
(4.12)

\[ \eta_\theta = \sum_{i=0}^{180} V_{f_i} \cos^4 \theta_i \]  
(4.13)

Equation 4.10 enables the calculation of \( V_f^* \) from the nominal value for \( V_f \) of 2 vol. %. The resulting values for \( E'_c \) are presented in Table 4-4. Although the three samples have different orientation factors, the effect on the overall modulus is small because \( V_f \) is low. The calculated Young’s modulus values will be compared to the measured Young’s modulus values in section 5.4.3, where strain-gauged specimens have been tested in tension to evaluate their self-healing ability.

<table>
<thead>
<tr>
<th></th>
<th>C6523 (T1/P2)</th>
<th>C6543 (T1/P1)</th>
<th>C6544C (T1/P1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \eta_\ell )</td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \eta_\theta )</td>
<td>0.51</td>
<td>0.37</td>
<td>0.31</td>
</tr>
<tr>
<td>( V_f )</td>
<td>0.02</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( V_f^* )</td>
<td>0.0102</td>
<td>0.0074</td>
<td>0.0062</td>
</tr>
<tr>
<td>( V_m )</td>
<td>0.98</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( E'_c ) (GPa)</td>
<td>13.15</td>
<td>13.04</td>
<td>12.99</td>
</tr>
</tbody>
</table>

### 4.5.2.3 Crack onset

The strain at first crack is calculated using equation 4.14 for ECC specimens in general and then specifically for C6523, C6543 and C6544C using a lower volume fraction of fibres which accounts for the fibre orientation. Based on ACK, the strain at first crack is given by:
\[ \varepsilon_{muc} = \left( \frac{12 \tau_o \gamma_m E_f V_f^2}{E_c E_m^2 R_f V_m} \right)^{\frac{1}{3}} \]  

(4.14)

For the ECC in the present work, the strain at first crack is estimated using \( V_f = 0.02 \) (and \( V_m = 0.98 \)), \( R_f = 20 \mu m \), \( \tau_o = 1.62 \) MPa, \( \gamma_m = \frac{G_d}{2} = 1.51 \) J.m\(^{-2}\), moduli values of \( E_f = 40 \) GPa, \( E_c = 13.54 \) GPa, \( E_m = 13 \) GPa. The values of the frictional bond strength \( \tau_o \) and chemical debonding energy \( G_d \) are obtained using the single fibre pull-out test data at 28 days (section 4.4.2). Hence, the strain at first crack for an ECC specimen is estimated at \( \varepsilon_{muc} \) of 0.00022, i.e. 0.022%.

The value of \( \gamma_m \) indicated above is relatively low compared to the value found in the literature for the matrix fracture energy rather than the interface fracture energy (Li, 1997; the matrix fracture energy being equal to 15 J.m\(^{-2}\); section 2.4.3). Hence, calculations were also carried out with \( \gamma_m = 15 \) J.m\(^{-2}\).

Table 4-5 presents the values of strain at first crack obtained using equation 4.14 with different values of \( V_f \) that consider the fibre orientation factor and the two values of fracture energy. The strain at first crack is estimated at 0.00030 for specimen C6523. Specimens C6543 and C6544C achieved values of 0.00024 and 0.00022 respectively. The results show that the specimen with a greater fibre orientation (a higher \( V_f^* \); C6523) has a higher strain at first crack (which could result in a higher stress at first crack). The resultant composite stress values at first crack obtained using Hooke’s Law (\( \sigma_{fc,thec.} \)) and the experimental values (\( \sigma_{fc,exp.} \)) from the specimens tested in tension are also presented in Table 4-5. Using different values of \( V_f \) and \( \gamma_m \), leads to the determination of a range of first cracking strains, \( \varepsilon_{muc} \), from 0.00011 to 0.00047.

The stress at first cracking obtained experimentally \( \sigma_{fc,exp} \) is 4.5 MPa for C6523 (\( V_f = 0.0102 \)), 3.3 MPa for C6543 (\( V_f = 0.0074 \)) and 2.3 MPa for C6544C (\( V_f = 0.0062 \)), the values are in line with the theoretical stress values at first crack obtained using \( \gamma_m \) equal to 15 J.m\(^{-2}\). Hence, \( \gamma_m = 15 \) J.m\(^{-2}\) will be used in further calculations.
Table 4-5: Values of strain at first crack obtained using the ACK equation and the resultant value of stress at first crack using Hooke’s Law. The highlighted values represent the ‘best’ agreement between model and experiment.

<table>
<thead>
<tr>
<th>$V_f$</th>
<th>$Y_m$ (J.m$^{-2}$)</th>
<th>$\varepsilon_{mu}$</th>
<th>$\sigma_{fc,\text{theo.}}$ (MPa)</th>
<th>$\sigma_{fc,\text{exp.}}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.02</td>
<td>15</td>
<td>0.00047</td>
<td>6.38</td>
<td>/</td>
</tr>
<tr>
<td>0.02</td>
<td>1.5</td>
<td>0.00022</td>
<td>2.96</td>
<td>/</td>
</tr>
<tr>
<td>0.0062 (C6544C)</td>
<td>15</td>
<td>0.00022</td>
<td>2.98</td>
<td>2.3</td>
</tr>
<tr>
<td>0.0062 (C6544C)</td>
<td>1.5</td>
<td>0.00010</td>
<td>1.36</td>
<td>2.3</td>
</tr>
<tr>
<td>0.0074 (C6543)</td>
<td>15</td>
<td>0.00024</td>
<td>3.28</td>
<td>3.3</td>
</tr>
<tr>
<td>0.0074 (C6543)</td>
<td>1.5</td>
<td>0.00011</td>
<td>1.52</td>
<td>3.3</td>
</tr>
<tr>
<td>0.0102 (C6523)</td>
<td>15</td>
<td>0.00030</td>
<td>4.06</td>
<td>4.5</td>
</tr>
<tr>
<td>0.0102 (C6523)</td>
<td>1.5</td>
<td>0.00014</td>
<td>1.89</td>
<td>4.5</td>
</tr>
</tbody>
</table>

However, as noted earlier, ACK has been developed for continuous aligned fibres. Tjiptobroto and Hansen (1993), as cited by Bentur and Mindess (2007), extended the ACK theory to short oriented fibres: equations 4.15 and 4.16. The first equation is based on the assumption that the stress transfer length is equal to the fibre length $L_f$ and that there is a linear strain distribution along the fibre:

$$\varepsilon_{mu} = \left( \frac{2Y_mV_m}{[(3/4)E_c-(7/24)E_fV_f^2(1+\alpha)]\alpha L_f} \right)^{1/2}$$ (4.15)

where:

$$\alpha = \frac{E_mV_m}{E_fV_f}$$ (4.16)

Using $V_m = 0.98$, $E_t = 40$ GPa, $L_f = 8$ mm, $Y_m = 15$ J m$^{-2}$, equation 4.15 enables the calculation of $\varepsilon_{mu}$ (considering $E_c$, $V_f^2$, and $\alpha$) as detailed in Table 4-6. The values obtained are slightly lower than the values shown in Table 4-5. This is perhaps not surprising as there is no physical basis for the assumption that the stress transfer length is equal to the fibre length, especially for long fibres.
Table 4-6: Values of strain at first crack based on the first model of Tjiptobroto and Hansen (1993) and obtained using $\gamma_m = 15 \text{ J m}^{-2}$

<table>
<thead>
<tr>
<th>Specimens</th>
<th>C6523</th>
<th>C6543</th>
<th>C6544C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_c$ (GPa)</td>
<td>13.54</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$V_f^*$</td>
<td>0.0102</td>
<td>0.0074</td>
<td>0.0062</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>15.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon_{\mu u}$</td>
<td>0.000168</td>
<td>0.000163</td>
<td>0.000161</td>
</tr>
</tbody>
</table>

The second equation assumes that the stress transfer length is smaller than the fibre length and that there is a linear strain distribution along the fibre:

$$\varepsilon_{\mu u} = \left(\frac{2\gamma_m V_f}{[(3/4)E_c - (7/24)E_f V_f^* (1+\alpha)\alpha(\beta l_f)]}\right)^{1/2}$$  \(4.17\)

where:

$$\beta = \frac{l_{tr}}{l_f/2}$$  \(4.18\)

and where:

$$l_{tr} = \frac{d_f \varepsilon_{\mu u c}(1+\alpha) E_f}{4\tau_0}$$  \(4.19\)

Equation 4.19 uses the value of strain at first crack obtained in equation 4.14, i.e. $\varepsilon_{\mu u c} = 0.00047$ for $\gamma_m = 15 \text{ J m}^{-2}$. Equation 4.14 enables the calculation of $\varepsilon_{\mu u}$ (considering $E_c$, $V_f^*$, $\alpha$, $l_{tr}$ and $\beta$) as detailed in Tables 4-7. The values of strain at first crack $\varepsilon_{\mu u}$ obtained are closer to those obtained using the ACK equation and the experimental values using Hooke’s Law, and are therefore more realistic. Overall, these calculations confirm the suitability of the ACK equation when using $\gamma_m = 15 \text{ J m}^{-2}$ and the appropriate fibre volume fraction $V_f^*$ taking into consideration the fibre orientation. In “second” position, the equation suggested by Tjiptobroto and Hansen (1993) when assuming that the stress transfer length is smaller than the fibre length and that there is a linear strain distribution along the fibre also gives good agreement. This is probably because it takes into consideration the effect of the frictional bond strength on both $\varepsilon_{\mu u c}$ and $l_{tr}$.
Table 4-7: Values of strain at first crack based on the second model of Tjiptobroto and Hansen (1993) and obtained using $\gamma_m = 15 \text{ J m}^{-2}$

<table>
<thead>
<tr>
<th>Specimens</th>
<th>C6523</th>
<th>C6543</th>
<th>C6544C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_c$ (Pa)</td>
<td>13540000000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$V_f^*$</td>
<td>0.0102</td>
<td>0.0074</td>
<td>0.0062</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>15.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon_{muc}$</td>
<td>0.00047</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$l_r$</td>
<td></td>
<td>0.0020</td>
<td></td>
</tr>
<tr>
<td>$\beta$</td>
<td></td>
<td>0.49</td>
<td></td>
</tr>
<tr>
<td>$\varepsilon_{mu}$</td>
<td>0.000240</td>
<td>0.000232</td>
<td>0.000229</td>
</tr>
</tbody>
</table>

4.5.2.4 Multiple cracking

i. Introduction

After the appearance of the initial crack, ECC exhibits multiple cracking under tensile stress, due to the transfer of load from the fibre to the matrix via the fibre-matrix interface away from the plane of a matrix crack, leading to the formation of further cracks. However this occurs on the condition that the fibre volume fraction exceeds $V_{f,crit}$. In this section, the critical fibre volume fraction models and a model for the behaviour of ECC material after the first crack will be considered.

ii. Critical volume fraction of fibres $V_{f,crit}$

For a fibre reinforced material to achieve pseudo-ductility under stress, it should contain the minimum volume of fibres, termed critical fibre volume fraction $V_{f,crit}$ to enable fibres to bridge the first crack, preventing catastrophic fracture. Equation 4.20 detailed in section 2.4.3 enables the calculation of $V_{f,crit}$ for a particular composition.

$$V_{f,crit} \geq \frac{\sigma_{mu}}{\sigma_{fu}}$$

(4.20)

For a typical ECC, using fibres with a tensile strength of 1560 MPa in a cementitious matrix having a tensile strength of 2.5 MPa for a water-cement ratio of 0.35 (Still, 2004), the minimum volume of reinforcement would be 0.16 %. Our current mixes use a fibre content of 2 vol. %, which is above the value calculated and this explains why bridging can occur and hence why first cracking does not lead to catastrophic failure. The higher the fibre tensile strength, the
lower the fraction of fibres required to bridge the cracks. However, this model is valid for aligned and continuous fibres and therefore for ECCs with fibres oriented in several directions, a greater amount of fibres would be necessary to compensate for fibre misalignment and discontinuity.

Bentur and Mindess (2007) suggested a modification to equation 4.20 for short and oriented fibres in equation 4.21:

\[ V_{f,\text{crit}} = \frac{\sigma_{mu}}{\eta_0 \tau_0 L_f / d_f} \]  

(4.21)

For a cementitious matrix having a tensile strength of 2.5 MPa, a fibre length of 8 mm, a fibre diameter of 20 µm, \( \tau_0 = 1.62 \) MPa, and values for \( \eta_0 \) of 0.51, 0.37 and 0.31 (as for specimens C6523, C6543 and C6544C respectively), the corresponding values of \( V_{f,\text{crit}} \) are 0.008, 0.010 and 0.012. The results mean that if a random fibre orientation is expected, then the critical volume fraction increases (to compensate for fibre orientation) in order for the fibres to bridge the cracks and lead to pseudo-ductility. For ECC material, where a random fibre orientation is expected, these values are still less than the volume fraction used (\( V_f = 0.02 \)) and so the material is expected to show pseudo-ductility.

Naaman (1987) describes the condition for achieving pseudo-ductile behaviour expressed in terms of \( V_{f,\text{crit}} \) using equation 4.22:

\[ V_{f,\text{crit}} = \frac{\sigma_{mu} d_f}{\tau_0 L_f \lambda - \alpha} \]  

(4.22)

Using the values of \( \tau_0 = 1.62 \) MPa, \( d_f = 20 \) µm, \( L_f = 8 \) mm, \( \sigma_{mu} = 2.5 \) MPa, and with the coefficients taken as \( \lambda = 0.30 \) and \( \alpha = 0.05 \) (Naaman, 1987), give \( V_{f,\text{crit}} \) a value of 0.015 which is closer to the value suggested by Li et al. (2002b). It should be noted that \( \lambda-\alpha \) could be viewed as a constant in equation 4.22.

The model suggested by Li (1997) enables the calculation of \( V_{f,\text{crit}} \) for ECCs based on equations 4.23, 4.24 and 4.25 as detailed in section 2.4.3:
\[ V_{f,\text{crit}} = \frac{12J_c}{g \tau_0 (\xi_f / d_f) \delta_o} \]  \hspace{1cm} (4.23)

\[ \delta_o = \frac{\tau_0 L_f^2}{E_f d_f (1+\eta)} \]  \hspace{1cm} (4.24)

\[ \eta = \frac{V_f E_f}{V_m E_m} \]  \hspace{1cm} (4.25)

Figure 4-15 compares the effect of frictional bond strength \( \tau_0 \) on \( V_{f,\text{crit}} \) using a crack tip toughness of 15 J m\(^{-2} \) using the work of Li (1997) and material from the current study. The work done by Li uses \( E_f = 120 \) GPa, \( L_f = 12.7 \) mm, \( d_f = 0.038 \) mm, \( g = 2.0 \), \( E_m = 25 \) GPa and our current work uses \( E_f = 40 \) GPa, \( L_f = 8 \) mm, \( d_f = 0.040 \) mm, \( g = 2.0 \), \( E_m = 13 \) GPa. Figure 4-15 shows that the critical volume fraction of fibres decreases with increasing bond strength. This probably means that for a specific matrix toughness, the less coating on the surface of the fibre, the higher the fibre matrix frictional bond strength and therefore the lower the critical volume fraction of fibres necessary to achieve pseudo-ductile behaviour under stress.

For the current ECCs having a fibre-matrix frictional bond strength of 1.62 MPa (see section 4.4.2), the critical volume fraction of fibres is 0.4 % compared with Li (1997) obtaining a similar value of \( V_{f,\text{crit}} \), but with a fibre-cement frictional bond strength of 1.2 MPa. Again, the conclusion is that the volume fraction used (2 vol. %) is sufficient for pseudo-ductility to occur.
iii. Behaviour after first crack

As mentioned in section 2.4.5, based on ACK stress transfer which considers continuous aligned fibres, the distance away \( x \) from the crack at which the cracking stress is reached (transfer length) is given in equation 4.26:

\[
x = \frac{V_m \sigma_{mu} R_f}{2 V_f \tau_0}
\]  

(4.26)

Using \( V_m = 0.98 \), \( R_f = 20 \, \mu \text{m} \), \( \tau_0 = 1.62 \, \text{MPa} \) and \( \sigma_{mu} = \sigma_{fc} = 3.3 \, \text{MPa} \) as the stress at first crack, result in the value \( x = 1 \, \text{mm} \). A low value of \( x \) is preferred as it is associated with a higher number of cracks and hence a greater strain and pseudo-ductility.

However, considering fibre orientation for ECCs and using \( V_f^* \), equation 4.26 can be modified to give equation 4.27:

\[
x^* = \frac{V_m \sigma_{mu} R_f}{2 V_f^* \tau_0}
\]  

(4.27)

The modified transfer lengths shown in Table 4-8 expressing \( x^* \) for C6523, C6543 and C6544C specimens which depend on their respective fibre volume fraction and the experimental stress.
at first crack, are larger than the value from equation 4.26. For example for a similar stress at first crack (3.3 MPa), the transfer length $x^*$ is 2.70 mm compared with a value of 1 mm based on continuous aligned fibres.

Table 4-8: Theoretical transfer length $x^*$ based on modified ACK model calculated using fibre orientation data

<table>
<thead>
<tr>
<th></th>
<th>C6523 (T1/P2)</th>
<th>C6543 (T1/P1)</th>
<th>C6544C (T1/P1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$V_f^*$</td>
<td>0.0102</td>
<td>0.0074</td>
<td>0.0062</td>
</tr>
<tr>
<td>$\sigma_{fc}$ (MPa)</td>
<td>4.5</td>
<td>3.3</td>
<td>2.3</td>
</tr>
<tr>
<td>$x^*$ (mm)</td>
<td>2.67</td>
<td>2.70</td>
<td>2.24</td>
</tr>
</tbody>
</table>

Wu and Li (1995) suggested an alternative modification to equation 4.26 considering random and discontinuous fibres:

$$x_d = \frac{L_f - \sqrt{L_f^2 - 2\pi L_f \varphi x}}{2}$$  \hspace{1cm} (4.28)

where $\varphi = \frac{4}{\pi g}$  \hspace{1cm} (4.29)

Using $L_f = 8$ mm, $g = 2$ and $x = 1$ mm, then $\varphi = 0.637$ and hence $x_d$ is 1.2 mm.

The result obtained with equation 4.28 signifies, as expected, that with random and discontinuous fibres, the composite has a greater transfer length $x_d$ (1.2 mm) compared with a composite made with aligned continuous fibres ($x = 1$ mm). However, the $x_d$ value remains lower than the values presented with the modified ACK model, although it seems that the modification of the ACK is more plausible as it simply considers that the fibres contribute less (hence a lower fibre volume fraction considered) rather than incorporating physical characteristics such as fibre length.

From these values and knowing that the saturation values of the crack spacing will be between one and two times the transfer length, enables the mean saturation crack spacing to be estimated as $x_f$ (equation 4.30):

$$x_f = \frac{3}{2} x$$  \hspace{1cm} (4.30)
Therefore, from equations 4.26, 4.27 and 4.28, the mean saturation crack spacing may be estimated for each model using equations 4.31, 4.32 and 4.33:

\[
X_f = \frac{3V_m \sigma_{mu} R_f}{4V_f \tau_o} \quad (4.31)
\]

\[
X_f^* = \frac{3V_m \sigma_{mu} R_f}{4V_f \tau_o} \quad (4.32)
\]

\[
X_{d,f} = x \frac{3(l_f - l_f^2 - 2\pi l_f \varphi x)}{4} \quad (4.33)
\]

Using the values \(x, x^*\) and \(x_d\) obtained with equations 4.26, 4.27 and 4.28 respectively, the expected average final crack spacing is expressed for each model \((x_f, x_f^*, x_{d,f})\) in Table 4-9.

**Table 4-9: Theoretical expected average final crack spacing for each model**

<table>
<thead>
<tr>
<th></th>
<th>C6523 (T1/P2)</th>
<th>C6543 (T1/P1)</th>
<th>C6544C (T1/P1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(V_m)</td>
<td>0.98</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(V_f)</td>
<td>0.02</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(V_f^*)</td>
<td>0.0102</td>
<td>0.0074</td>
<td>0.0062</td>
</tr>
<tr>
<td>(\sigma_{fc}) (MPa)</td>
<td>4.5</td>
<td>3.3</td>
<td>2.3</td>
</tr>
<tr>
<td>(x) (mm)</td>
<td>1.00</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(x^*) (mm)</td>
<td>2.67</td>
<td>2.7</td>
<td>2.24</td>
</tr>
<tr>
<td>(x_d) (mm)</td>
<td>1.20</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(x_f) (mm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(x_{d,f})</td>
<td>1.80</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 4-16 shows the local crack spacings observed at different locations along the specimen length for specimen C6523 and, as an inset, the specimen tested which exhibits (blocks of) areas with cracks and areas without cracks. This is probably due to the inhomogeneity in the ECC microstructure. The graph reveals that most of the crack spacing remains below 5 mm and counting only the crack spacing within the “block” of cracks, the average value is 3.18 mm. Specimen C6523 presents 31 cracks over a length of 177 mm, taking the crack density to 0.17 crack/mm. Comparing these observations with the theoretical values calculated, it appears that the experimental value (3.18 mm) is comparable with the derived values and closest to
the result from the modified ACK model considering fibre orientation (i.e. 4.01 mm). However, the value obtained is also comparable to the transfer length value calculated (2.67 mm) using the modified ACK model considering fibre orientation. Whilst considering the mean crack spacing calculated, these seem to be lower than the mean crack spacing observed. In the regions where there is extensive cracking, the measured spacings will be of the same order as the calculated values. The conclusion is that final failure intervenes before saturation cracking is developed in the un-cracked regions. Hence, equation 4.29 could be used to predict the crack spacing between “areas” of crack knowing the tensile cracking stress of the material (at any time since the material cracks at constant stress) when the material is used in a structure.

Similarly to specimen C6523, Figure 4-17 shows the composite crack spacing observed as a function of the specimen length for specimen C6543. The average crack spacing over the specimen had a value of 3.56 mm and a crack spacing of 2.83 mm was observed within the “blocks” of cracks. The experimental value of $x^*$ of 2.83 mm is again closest to the modified ACK model with a theoretical value of 2.70 mm and confirms equation 4.29 in the cracked regions. Specimen C6543 presents 59 cracks over a length of 206 mm and, hence the crack density is 0.28 crack/mm. Comparing Figures 4-16 and 4-17, it appears that specimen C6543 presents a lower crack spacing and a higher crack density although specimen C6523 presents a higher $V_f^*$ and ultimate strain. Possibly, this is due to either the presence of non-visible fine cracks or higher crack widths for specimen C6523 compared with specimen C6543 promoting a higher strain.

The comparison of Figures 4-16 and 4-17 shows that the modified ACK model gives a reasonable description of the observed behaviour.
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Figure 4-16: Plot showing the variation of local crack spacing along the gauge length of specimen C6523 after tensile testing to failure, which shows the crack spacing observed and averaged (complete full specimen and within blocks of cracks)

Figure 4-17: Plot showing the variation of local crack spacing along the gauge length of specimen C6543 after tensile testing to failure, which shows the crack spacing observed and averaged (complete full specimen and within blocks of cracks)
iv. Discussion

Overall, the analysis in this section has shown that ACK theory, using a modified fibre volume fraction considering fibre orientation, gives a reasonable description of crack onset behaviour and the subsequent range of crack spacings achieved in the ECC material. Within this discussion, the comparison between idealised ACK behaviour and the response of the ECC material is considered further.

Figures 4-18 and 4-19 show classic ACK behaviour and a typical response of the ECC material tested in the current work. Figure 4-18 shows an elastic region, a short plateau in which matrix cracking occurs and then, a second elastic region in which the stiffness is controlled by the bridging fibres. From Figure 4-19, it can be seen that the ECC exhibits the elastic region, followed by multiple cracking, with the cracks occurring at more or less constant stress, as expected from ACK. The third region of the ACK is not shown by the ECC material tested here. It is suggested that this is because premature failure of the ECC material occurs before matrix cracking is saturated throughout the specimen. The failure most likely occurs as a result of fibre pull-out, which will be considered further in the next section where alternative strength models based on fibre fracture and pull-out are considered.

ACK theory supposes that once initial cracking occurs at a specific stress, then the next phase of cracking occurs at constant stress in the multiple-cracking zone and, ECC material shows cracking at a constant stress. This reveals the suitability of the ACK model for ECC material. When a matrix crack occurs under displacement controlled loading, this leads to a reduction in the specimen stiffness and hence there is a load drop. Therefore, the load needs to be increased in order to cause further matrix cracking to occur. In an idealised material, this would lead to a “saw-tooth” response in the stress-strain behaviour as the number of matrix cracks increases, which is similar to the behaviour seen. With regard to the final failure, the local fibre or pull-out controlled strength will be different at the various crack sites and will depend on the bridging fibre orientation and embedded lengths. It is not possible to be definitive as to whether the final failure occurs at the site of the first crack or occurs at a subsequent crack location. One interesting observation is that with fibres of length of 8 mm, the maximum pull-out length cannot exceed 4 mm. In a gauge length of 200 mm, a displacement of 4 mm (necessary to pull the fibres out) would correspond to a tensile strain
of 2%. This number is comparable to the ductility shown by the material and so perhaps provides further evidence that failure is controlled by pull-out.

In the next section, models for strength are considered in more detail.

![Diagram 1](image1.png)

**Figure 4-18:** Typical experimental and theoretical stress-strain curve, according to the ACK theory (Blom et al., 2008)

![Diagram 2](image2.png)

**Figure 4-19:** Tensile stress-strain results of ECC material
4.5.3 Ultimate strength model

4.5.3.1 Introduction

Experimental observations of ECCs showed that composite failure occurs after multiple cracking, due to fibre matrix debonding and some evidence of limited fibre fracture. In this section, simple estimates of the strength of ECCs based on two models are presented: one where fibre fracture controls the final failure and the other where fibre pull-out is the controlling phenomenon.

4.5.3.2 Strength controlled by fibre fracture

Equation 4.34, as detailed in section 2.4.4, is used to estimate the composite maximum strength after initial matrix failure and considering that it is only the fibres that support the load in the composite.

\[ \sigma_{c,max} = \eta_l \eta_\theta V_f \sigma_{fu} \] (4.34)

Equations 4.35, 4.36 and 4.37 enable the calculation of \( \eta_l \) and \( \eta_\theta \) (see section 2.4.4):

\[ \eta_l = 1 - \left[ \frac{\tanh \left( \frac{\beta L_f}{2} \right)}{\left( \frac{\beta L_f}{2} \right)} \right] \] (4.35)

\[ \beta = \frac{2 \pi G_m}{[E_f A_f \ln \left( \frac{R}{R_f} \right)]} \] (4.36)

\[ \eta_\theta = \sum_{i=0}^{i=180^\circ} V_{f_i} \cos^4 \theta_i \] (4.37)

Equation 4.34 is a function of the fibre characteristics such as fibre orientation characterised by \( \eta_\theta \). In section 4.3.2, data on fibre orientations obtained were used to calculate \( \eta_\theta \) for each specimen, which lead to a value for \( \eta_\theta \) of 0.51 for specimen C6523, while the values for the other two specimens, C6543 and C6544C were 0.37 and 0.31 respectively.

It will be recalled that the nominal volume fraction for all the materials in the present work is 2 %. Hence, calculating the strength for each system using equation 4.34 shows that the values vary from 9.6 MPa (C6544C), to 11.6 MPa (C6543) and up to 15.8 MPa (C6523) for a more aligned system. These values are higher than the strengths achieved in practice, which do not
exceed 5 MPa (see Figure 4.19). Furthermore, the experimental strength of the more aligned system is not significantly higher. These calculations confirm the capacity of the fibres to bridge the matrix cracks, but the over-estimate of strength suggests that a model based on fibre pull-out from a frictionally bonded interface is likely to be more appropriate.

4.5.3.3 Strength controlled by fibre pull-out

For a nominal fibre length of 8 mm, the mean fibre pull-out length $l_{po}$ is 2 mm, independent of the fibre orientation. Hence, the corresponding stress on the composite to initiate pull-out failure can be calculated from equations 4.38 and 4.39, see section 2.4.4. Relation 4.38 enables the calculation of the load to pull-out a single fibre and relation 4.39, the stress on the composite to initiate pull-out of the fibre.

$$P = \pi d_f l_{po} \tau_o$$  \hspace{1cm} (4.38)

$$\sigma_{po} = V_f \frac{P}{\pi R_f^2} = \frac{4V_f \tau_o l_{po}}{d_f}$$  \hspace{1cm} (4.39)

The fibre diameter is 40 µm and the frictional shear strength $\tau_o$ is taken as 1.62 MPa, which is consistent with the literature (Li, 2003) and measurements made as part of the present study using a single fibre pull-out specimen (see section 4.4.2). Substitution into equation 4.39 gives a strength of 6 MPa. Assuming that the fibre orientation modifies the strength by pull-out in a similar way to the strength when controlled by fibre failure, then the predicted strengths for systems that fail by pull-out are in the range 1.9 – 3 MPa. In fact, using the same orientation factors for pull-out and fibre controlled failures is unlikely to be appropriate. Fibres with greater misalignment are likely to contribute more to pull-out strength than the fibre-based strength and hence the range 1.9 – 3 MPa is likely to be a lower bound, while 6 MPa will represent an upper bound. Overall the results from the pull-out model are consistent with the experimental data, notwithstanding the simplifications made such as ignoring the random nature of the fibre distribution and the role of defects such as porosity.

Noting also that based on considerations of fibre pull-out, the critical opening displacement of the crack that controls fracture should be of the order of the mean pull-out length. Based on a specimen gauge length of 200 mm, this gives a nominal strain of 1 %, which is around half
of the total strain observed in a typical specimen after first crack. The balance of the strain is presumably associated with the opening of the other cracks.

4.5.4 Summary
To summarise, the study reveals that the ACK model is well adapted for ECCs on the condition that fibre orientation is considered: a modified ACK model gives a reasonable representation of the crack onset behaviour. Although stage 3 corresponding to the increase of stress with strain observed in the ACK model does not exist for ECCs, stage 2 corresponding to multiple cracking at constant stress seems to perfectly fit ECCs. The behaviour of ECCs after the first crack demonstrates that cracks appear at constant stress due to the low volume fraction of fibres. The crack spacings observed seem lower than those calculated; a model has been defined for ECCs to estimate the crack spacing based on the cracking stress of the composite. The strength model demonstrated that fibre pull-out is more likely to control the composite strength rather than fibre fracture. Fibre fracture seems to govern first cracking and fibre pull-out, multiple cracking.

4.6 Flexure testing

4.6.1 Introduction
Although the tensile test results showed some variability from sample to sample, they provided valuable information as to the mechanical performance of ECCs and the influence of several material and processing parameters. In this section, the flexural testing results are presented with a view to gaining further understanding of the effect of fibre and process type on the mechanical performance.

4.6.2 Elastic-plastic behaviour and multiple fine cracks
Figure 4-20 shows a photograph of a portion of a specimen tested in flexure. The image indicates the presence of multiple fine cracks in the vicinity of a major crack associated with the failure of the specimen. This is representative of most of the specimens tested and shows the behaviour of the material under a flexural stress: the material exhibits multiple fine cracks and then one of the cracks widens leading to the failure of the specimen in a similar way to the specimens tested in tension.
The mechanical data obtained from a specimen tested in flexure are plotted as bending moment against the curvature (Figure 4-21). The bending moment-curvature graph is usually used to characterise the non-linearity of concrete in the strain-softening region. The curvature is determined based on the strain at the bottom and top probes. The larger the area below the curve, the greater the apparent ‘toughness’ of the specimen and the more ‘graceful’ the failure.

Looking in more detail, the moment-curvature graph (Figure 4-21) shows for an ECC specimen, an initial elastic response region up to the formation of the first crack, which is followed by a region of pseudo-plasticity, associated with the formation of multiple cracks, before the final failure of the specimen. As a comparison, the specimen without fibres used as a control presents only an elastic region as expected.

According to Figure 4-21, the bending moment at which first crack occurs equals to $M_{fc} = 1.0135\text{ MN.mm}$. The bending stress at first crack can be calculated from the bending moment $M_{fc}$ using the equation:

$$\sigma_{b,fc} = \frac{M_{fc}y}{I} \quad (4.40)$$

Where $I = \frac{d_1 d_2^3}{12} \quad (4.41)$
And \( y = \frac{d_2}{2} \) \hspace{1cm} (4.42)

Using \( d_1 = 100 \text{ mm} \) and \( d_2 = 100 \text{ mm} \), the derived bending stress at first crack \( \sigma_{b,fc} \) is 6.1 MPa. This value is twice the value of stress at first crack of the specimens tested in tension with approximately a value of 3 MPa. This is not surprising because in flexure, the peak stress is confined to the surface whereas in tension, the stress is throughout the bulk of the specimen. The larger first crack stress value obtained in flexure compared with the tensile value is also probably due to the larger size of the specimen tested in flexure and the extreme sensitivity of the tensile testing. Such size/mode of loading effects are well documented in brittle materials.

![Figure 4-21: Typical bending moment – curvature response for a beam specimen tested in four-point bending at 27 days (C2701: made with T2 following P1)](image)

Flexural performance at 27 days of specimens made with different fibre and process types (Table 4-10) are compared in the next section. As a control, specimens without fibres (C2961, C2962 and C2963) were manufactured and tested at the same age:
Table 4-10: Flexure experimental programme

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Description</th>
<th>Age when tested</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(Fibre type/Process)</td>
<td>(days)</td>
</tr>
<tr>
<td>C2701</td>
<td>T2/P1</td>
<td>27</td>
</tr>
<tr>
<td>C2702</td>
<td>T2/P1</td>
<td>27</td>
</tr>
<tr>
<td>C2734</td>
<td>T1/P1</td>
<td>27</td>
</tr>
<tr>
<td>C2736</td>
<td>T1/P1</td>
<td>27</td>
</tr>
<tr>
<td>C2747</td>
<td>T2/P2</td>
<td>27</td>
</tr>
<tr>
<td>C2749</td>
<td>T2/P2</td>
<td>27</td>
</tr>
<tr>
<td>C2788</td>
<td>T1/P2</td>
<td>27</td>
</tr>
<tr>
<td>C2790</td>
<td>T1/P2</td>
<td>27</td>
</tr>
<tr>
<td>C2961</td>
<td>No fibres – Control specimen</td>
<td>27</td>
</tr>
<tr>
<td>C2962</td>
<td>No fibres – Control specimen</td>
<td>27</td>
</tr>
<tr>
<td>C2963</td>
<td>No fibres – Control specimen</td>
<td>27</td>
</tr>
</tbody>
</table>

4.6.3 Effect of the fibre type

Figure 4-22 presents the results obtained for specimens without fibres aged for 27 days. In contrast to the specimens containing fibres, specimens without fibres present only an elastic region followed by a sudden failure of the specimens. Failure of the specimens is characterised by a single major crack. This is essentially the same behaviour as seen for specimens without fibres tested in tension (section 4.2.2.3).

Figure 4-22: Bending moment-curvature data for specimens tested in four point bending at 27 days: without fibres as a control

Figures 4-23 and 4-24 present the results obtained for specimens made with two different fibre types and using Process P1 (fibres added prior to water) and Process P2 (fibres added...
after water addition) respectively. Although some variation is noticed as to the bending moment value at the first crack, the specimens exhibit a broadly similar response.

Comparing the results for the two fibre types when process 1 (P1) is used (Figure 4-23), both specimens made with fibre type T2 exhibit a high maximum curvature (up to $7 \times 10^{-4}$ mm$^{-1}$) compared with specimens made with type T1 fibres, where the value of curvature is approximately $3 \times 10^{-4}$ mm$^{-1}$. A higher deflection at failure and maximum moment load are measured for the specimens made with T2 fibres. The difference in mechanical performance between the two mixes is likely to be due to the ability of the fibres to pull-out in the tensile region of the specimen. As seen in section 3.2.2, fibres type T2 are of a “resin-bundled type” and are therefore stiffer, and could be more suitable for Process P1, as specified by the supplier. This additional treatment could improve the quality of their dispersion in the cementitious matrix with Process P1. On the other hand, fibres type T1 could either present a poor dispersion and agglomerate or may not be as well oriented in the region subjected to the greatest stress. A similar comparison is conducted for the two fibre types, but with Process P2 (Figure 4-24). The results are similar for the specimens tested although fibre type T2 showed a slight advantage over fibre type T1, hence a definite conclusion cannot be made at this stage.

Figure 4-23: Bending moment – curvature data for specimens tested in four point bending at 27 days: effect of fibre type (using Process P1)
4.6.4 Effect of the process

As discussed in section 3.3.1, two processes (mixing) methodologies have been considered. Having determined that fibre type T2 leads to a greater pseudo-ductility, these were used to compare the two different processes, and the results are presented in Figure 4-25.

As stated in section 4.6.3, specimens made from fibre type T2 and manufactured using Process P1 exhibit a curvature at failure of up to $7 \times 10^{-4} \text{ mm}^{-1}$. Specimens made with Process P2 give results that are more variable than those seen for Process P1 but show an increase in bending moment at failure and a reduction in curvature (an average of $2 \times 10^{-4} \text{ mm}^{-1}$ for the two specimens). Possibly fibre type T2 which are stiffer, are not suitable for Process P2 (but rather for Process P1 as suggested by the Supplier) as probably the resin-bundling promote a lower fibre dispersion meaning they remain as bundles with Process P2. However it has to be noted that Process P2 is preferred as enabling a better workability and ease of placement of the fresh mixture and the results in tensile testing suggest a better mechanical performance when Process P2 is used with fibre type T1 (section 4.2.3).

Further investigation, including fibre dispersion on cut sections of these specimens, would
enable a better understanding of the differences in mechanical performance. Furthermore, more data along with statistical analysis is required to make a definite conclusion.

![Bending moment-curvature data for specimens tested in four point bending at 27 days: effect of process type (using fibre Type T2)](image)

Figure 4-25: Bending moment-curvature data for specimens tested in four point bending at 27 days: effect of process type (using fibre Type T2)

### 4.7 Correlation of Mechanical Behaviour in Tension and Flexure with Physical Properties

#### 4.7.1 Introduction

In concrete technology, a high density and a low porosity are preferred as they are associated with a higher strength as the pores are seen as potential points of rupture of the material under stress. However, for the ECCs, where the load at failure is less important and the toughness is more relevant to its application, the optimum physical properties could be slightly different. In this section, the density and porosity values for each specimen tested in tension and flexure are identified. An attempt to correlate the physical properties with the mechanical performance of the ECCs and hence with the composition and manufacturing process is presented.

#### 4.7.2 Tensile test parameters

The tensile testing results showed that the thicker material is capable of pseudo-ductility, although variability in the mechanical performance remains, even between specimens from a
similar process and fibre type. In order to understand the variability in mechanical properties, it is important to appreciate and understand the relation between these properties and certain physical properties of the manufactured samples. Density and porosity measurements were made using samples cut from the tensile specimens, enabling a direct comparison of the mechanical performance with the physical properties.

Values of density and porosity are reported for a range of samples and compared with the mechanical properties in Table 4-11. The average density measured on twelve specimens was 1947 kg.m\(^{-3}\) with a standard deviation of 23 kg.m\(^{-3}\). The measured values of open porosity were on average 6.2 % with a standard deviation of 1.7 %, although there is no measure of scale (large number of small pores or small number of larger pores) or distribution (clustered, evenly dispersed, predominantly found at external faces of the specimen or at fibre/matrix interfaces). As a comparison, the control specimens (which do not contain fibres) present an average density value of 2072 kg.m\(^{-3}\) with a standard deviation of 4 kg.m\(^{-3}\), whilst the porosity, an average value of 4.5 % with a standard deviation of 0.3 %.

Table 4-11 shows that the specimens moulded and with the largest ultimate strains (\(\varepsilon\)): specimens C6523 and C6543 have also the highest porosities, 7.8 % and 9.5 %, which is consistent with the literature (Wang and Li, 2004). The highest density specimen with a high porosity (specimen C6523) shows a large strain after first crack and a high maximum stress. The highest porosity specimen (specimen C6543) associated with an average density presents the highest strain after first crack, but a lower maximum stress. The lowest density specimens (cast in slabs) are generally associated with a low maximum peak stress (\(\sigma_{\text{max}}\)) and also a low plastic strain \(\varepsilon\) from first crack, so giving a very low pseudo-ductility. In order to have a better idea of the relationships between the physical properties and mechanical performance of ECC materials, the data in Table 4-11 are plotted in Figures 4-26 and 4-27. The results reveal that the tensile strain \(\varepsilon\) increases with the porosity, however the density remains quite constant. On the other hand, the maximum stress \(\sigma_{\text{max}}\) slightly increases with the density, whereas the porosity remains quite constant, thus confirming the findings presented earlier in this paragraph. The graphical representation of the results also show that the specimens cast in a slab are likely to present a different behaviour to the ones cast in a mould; they do not follow the observed trend. The slab specimens present both a low tensile strain and a low maximum
stress associated with a high porosity and a low density, suggesting a possible issue with the compaction of the material during the manufacturing process. This reveals a possible combining effect when considering both density and porosity in order to achieve both a high tensile strain and maximum stress revealing the toughness of the ECC material.

Figure 4-26: Tensile strain $\varepsilon$ relationship with the porosity and density – the data circled correspond to the specimens cast in a slab
Although there is this variation, the data are consistent with the idea that a high porosity promotes a large strain after the initial crack and also a first crack at a lower stress whereas a high density, leads to a high maximum stress. A first crack at lower stress in high porosity specimens could be explained by the possibility that porosity may be indicative of a maximum initial flaw present (Wang and Li, 2004). If so, then it seems reasonable that higher levels of porosity promote first cracking and multiple cracking at lower loads. Whilst the trend is not conclusive, it appears that the ECCs can still operate (at least on a short term basis) despite the presence of significant porosity. The variability in the physical properties (especially porosity) and mechanical performance of specimens made from the same batch are relatively important and therefore conclusions on the effect of the process and fibre type on the physical properties are not possible at this stage. Although the variation in mechanical performance is consistent with the physical properties measured and in fact with cementitious materials in general, a way has to be found to control these physical properties to make the mechanical performance more consistent if this material is to be used in challenging commercial situations.
Table 4-11: Tensile test parameters with the corresponding values of density and porosity

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fibre / Process (Types)</th>
<th>Workability (mm)</th>
<th>Casting</th>
<th>Age tested (days)</th>
<th>$\sigma_{at\ FC}$ (MPa)</th>
<th>$\sigma_{max}$ (MPa)</th>
<th>$\epsilon$ from FC (%)</th>
<th>Density (kg.m$^{-3}$)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C6256</td>
<td>T2/P1</td>
<td>175 mm</td>
<td>mould</td>
<td>27</td>
<td>4.3</td>
<td>4.3</td>
<td>0.37</td>
<td>1951</td>
<td>4.6</td>
</tr>
<tr>
<td>C6257</td>
<td>T2/P1</td>
<td></td>
<td></td>
<td></td>
<td>4.3</td>
<td>4.4</td>
<td>0.95</td>
<td>1940</td>
<td>4.9</td>
</tr>
<tr>
<td>C6258</td>
<td>T2/P1</td>
<td></td>
<td></td>
<td></td>
<td>3.7</td>
<td>3.9</td>
<td>0.53</td>
<td>1946</td>
<td>5.1</td>
</tr>
<tr>
<td>C6522</td>
<td>T1/P2</td>
<td></td>
<td></td>
<td></td>
<td>4.5</td>
<td>4.7</td>
<td>2.17</td>
<td>1969</td>
<td>7.8</td>
</tr>
<tr>
<td>C6523</td>
<td>T1/P2</td>
<td></td>
<td></td>
<td></td>
<td>4.5</td>
<td>4.9</td>
<td>1.71</td>
<td>1969</td>
<td>7.8</td>
</tr>
<tr>
<td>C6524</td>
<td>T1/P2</td>
<td>208.5 mm</td>
<td>mould</td>
<td>27</td>
<td>3.9</td>
<td>4.7</td>
<td>1.41</td>
<td>1987</td>
<td>5.4</td>
</tr>
<tr>
<td>C6541</td>
<td>T1/P1</td>
<td></td>
<td></td>
<td></td>
<td>3.9</td>
<td>4.3</td>
<td>1.04</td>
<td>1937</td>
<td>4.5</td>
</tr>
<tr>
<td>C6542</td>
<td>T1/P1</td>
<td></td>
<td></td>
<td></td>
<td>3.7</td>
<td>3.8</td>
<td>0.33</td>
<td>1962</td>
<td>4.6</td>
</tr>
<tr>
<td>C6543</td>
<td>T1/P1</td>
<td>175 mm</td>
<td>mould</td>
<td>27</td>
<td>3.3</td>
<td>3.9</td>
<td>2.24</td>
<td>1952</td>
<td>9.5</td>
</tr>
<tr>
<td>C6544A</td>
<td>T1/P1</td>
<td></td>
<td></td>
<td></td>
<td>2.4</td>
<td>2.5</td>
<td>0.28</td>
<td>1915</td>
<td>7.8</td>
</tr>
<tr>
<td>C6544B</td>
<td>T1/P1</td>
<td></td>
<td></td>
<td></td>
<td>2.2</td>
<td>2.2</td>
<td>0.16</td>
<td>1913</td>
<td>7.6</td>
</tr>
<tr>
<td>C6544C</td>
<td>T1/P1</td>
<td>172 mm</td>
<td>slab</td>
<td>27</td>
<td>2.3</td>
<td>2.3</td>
<td>0.13</td>
<td>1925</td>
<td>7.1</td>
</tr>
<tr>
<td>C7097</td>
<td>Control specimens – without fibres</td>
<td>/</td>
<td>mould</td>
<td>27</td>
<td>1.9</td>
<td>1.9</td>
<td>0.00</td>
<td>2074</td>
<td>4.4</td>
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<td>C7098</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.6</td>
<td>1.6</td>
<td>0.00</td>
<td>2082</td>
<td>4.3</td>
</tr>
<tr>
<td>C7099</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.8</td>
<td>2.8</td>
<td>0.00</td>
<td>2061</td>
<td>4.8</td>
</tr>
</tbody>
</table>

### 4.7.3 Flexural test parameters

In this section, values of density and porosity are reported for a range of samples and compared with the mechanical properties in flexure. Table 4-12 shows data for the flexural test specimens. The average density measured on eight specimens was 1862 kg.m$^{-3}$ with a standard deviation of 37 kg.m$^{-3}$. The porosity values measured on four samples were reasonably consistent, in the range of 1.1 – 1.6 %, although there is no measure of scale (large number of small pores or small number of again larger pores) or distribution (clustered, predominantly found at faces, evenly dispersed).

For the ECC performance in flexure, the important parameter to take into consideration is the curvature associated with the maximum load, characterising the toughness of the material under load. Regrouping the results obtained, the first observation is that the specimen having the highest density (1918 kg.m$^{-3}$) is also the one presenting the greatest maximum load (39.9 kN); however the one with a greater curvature has a density of 1887 kg.m$^{-3}$. The results demonstrate that for the ECC, there is a compromise between maximum load and curvature obtained. Indeed, the performance of specimens C2701 and C2702 are in general enhanced although the maximum load is not the highest. The table also shows that the specimens with a low density, such as C2736 and C2790, do not present an especially high curvature when tested nor a high maximum load when compared with the other specimens.
Table 4-12: Flexural test parameters with the corresponding values of density and porosity

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Description (Fibre type/Process)</th>
<th>Maximum load (ML) (kN)</th>
<th>Curvature at ML (x10^-4:mm^-1)</th>
<th>Density (kg.m^-3)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C2701</td>
<td>T2/P1</td>
<td>35.4</td>
<td>6.3</td>
<td>1887</td>
<td>1.5</td>
</tr>
<tr>
<td>C2702</td>
<td>T2/P1</td>
<td>32.9</td>
<td>5.6</td>
<td>1838</td>
<td>1.5</td>
</tr>
<tr>
<td>C2734</td>
<td>T1/P1</td>
<td>29.4</td>
<td>3.1</td>
<td>1848</td>
<td>/</td>
</tr>
<tr>
<td>C2736</td>
<td>T1/P1</td>
<td>27.8</td>
<td>1</td>
<td>1820</td>
<td>/</td>
</tr>
<tr>
<td>C2747</td>
<td>T2/P2</td>
<td>36.8</td>
<td>2.3</td>
<td>1905</td>
<td>1.6</td>
</tr>
<tr>
<td>C2749</td>
<td>T2/P2</td>
<td>39.9</td>
<td>1</td>
<td>1918</td>
<td>1.1</td>
</tr>
<tr>
<td>C2788</td>
<td>T1/P2</td>
<td>35.4</td>
<td>1.6</td>
<td>1857</td>
<td>/</td>
</tr>
<tr>
<td>C2790</td>
<td>T1/P2</td>
<td>20.9</td>
<td>0.5</td>
<td>1826</td>
<td>/</td>
</tr>
</tbody>
</table>

The results suggest overall that the curvature and density may well be correlated. The most important parameter for the ECC is the curvature associated with a maximum load which should not be too low in value (greater than 30 kN). The maximum load could be correlated with the density, which should be lower than approximately 1900 kg.m^-3. If the maximum load was high but the curvature low, it would represent a brittle material. The density should also be greater than approximately 1860 kg.m^-3; otherwise the value of both the curvature and the maximum load will be low. A compromise has to be made to obtain a significant curvature, determining the ductility of the ECC under load.

In an attempt at correlating the results with the composition and manufacturing process, the results revealed that specimens made with fibre T2 and Process P1 seem to exhibit better mechanical performance. However, more data and further investigation are required to confirm any meaningful conclusions at this stage.

### 4.8 Summary

Following the literature review, an investigation into two types of fibres and mixing processes was conducted in order to evaluate the mechanical performance of the ECCs. The tensile testing results demonstrated that the ECC specimens tested in tension exhibit multiple fine cracks associated with a certain degree of pseudo-ductility. Process P2 is preferred as it provides improved workability and the study reveals that fibre type T1 would be more suitable. Specimens moulded perform relatively well compared with machined specimens. The increase in specimen thickness compared with previous studies shows a decrease in the tensile strain reaching a plateau. The fractographic analysis revealed signs of a fibre-cement interaction.
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The fibre dispersion analysis at different scales shows an excellent fibre distribution for both processes. The fibre orientation of three specimens (two moulded and one machined) reveals a greater fibre alignment along the tensile axis in line with better mechanical performance (ultimate strain and maximum stress). This could explain the difference in mechanical performance observed between thicker specimens where it more likely to observe a random orientation of the fibres and thinner specimens (with a greater fibre alignment as seen in the literature). Therefore, thicker sections such as the one tested in this study (30 mm) may require either a higher $V_f$ or a fibre alignment in a specific direction to exhibit enhanced mechanical performance.

The single fibre pull-out testing data at 28 days of fibres embedded in cement enabled the determination of interfacial properties of ECCs. Valuable interfacial parameters such as the interfacial mean strength $\tau_d$, the chemical debonding energy $G_d$ and the frictional bond strength $\tau_0$ are characterised. This confirms the tailoring of the current fibres for a cementitious matrix to produce a pseudo-ductile behaviour of ECCs under tensile stress, as suggested in the literature review.

Theoretical models when applied to ECCs show that the ACK theory is in line with ECCs behaviour when a lower fibre volume fraction is considered and taking into consideration fibre orientation and that similarly to ACK, ECC present multiple cracking at constant stress. The fibre volume fraction confirms the suitability of using 2 vol. % fibres. The crack spacing observed between blocks of cracks is in line with the crack spacing calculated using the modified ACK model considering fibre orientation and can be used to predict crack spacing knowing the cracking stress of the composite. The strength model demonstrated that fibre pull-out is more likely to control the composite strength rather than fibre fracture. The application of existing theoretical models to ECC enables a better understanding of ECC and hence a contribution to the current body of knowledge.

Based on the flexural testing results, Process P1 and fibre T2 give improved mechanical performance of the ECCs, which is demonstrated by a high strain-deflection, associated with a high maximum bending moment. Process P2 is beneficial when used with fibre T1, and has also advantages in increasing the workability of the mixture in its fresh state.
consequence, Process P2 and fibre T1 are selected for future mixes.

The analysis of density and porosity of specimens tested in tension and flexure revealed that ECCs can perform well despite the presence of a high level of porosity and the best performance observed in tension corresponds to the specimen with the highest porosity. In particular, specimens with a high porosity exhibit a higher ultimate strain whereas the ones with a high density, a higher maximum stress, although the trend is not conclusive.

In the following chapter, the long-term durability is investigated, in the light of the mechanical properties presented in this chapter.
5 Durability

5.1 Introduction

In the previous chapter, the mechanical performance of ECCs was discussed including a correlation of the physical properties with the mechanical behaviour in tension and flexure. However, this is only one aspect of the behaviour of the material that must be characterised. The long-term durability is also important as the material should be able to exhibit pseudo-ductility for its whole design life, which is likely to be of the order of 150 years, despite the possible degradation of the material over time and changes in properties due to ageing. A further design concept of the durability of the material is its ability to cope with damage (cracking) when it occurs, even if this is comparatively early in its use. On this basis, there are three areas of interest that must be evaluated:

1. Natural ageing of the material. In addition to the potential damage due to external factors as detailed in section 2.6.2 and the deterioration due to cracking, cementitious materials are known to continue to harden as they age and as a consequence, increase in strength. This may impact on the ability of the material to behave in a pseudo-ductile way under stress, as this behaviour is dependent on the balance of properties between the fibre and the matrix.

2. Changes of the fibre-matrix interface. As discussed previously, the coating that is applied to the fibres has been tailored to control fibre slippage when the material is under stress, promoting pseudo-ductility and multiple cracking. However, surface layers are prone to attack by water and/or components present in the matrix solution. A change in the balance of properties of bonding between the fibres and the matrix may compromise the ability of the material to achieve multiple cracks. Hence, the ageing process at the fibre-cement interface may affect the pseudo-ductile behaviour of the ECC material over time.

3. Self-healing. Whereas the other two areas of interest deal with a change in properties, this third area considers the ability of the material to recover from damage on its own. The possible mechanism of healing within ECC material was discussed in section 2.6.5. The ability of the material to continue to self-heal throughout its lifetime and its limitations must be understood.
The current chapter presents the results of investigations into these three areas of interest, and summarises the key-findings. In order to assess the durability of the material, several lines of enquiry have been followed. Specimens were tested in flexure to evaluate the change in mechanical properties with time. The fibre surface chemistry was investigated with time including an attempt at evaluating the interface chemistry. The evolution of the fibre-cement interface property with time (up to 5 months) was also investigated through single fibre pull-out testing. Specimens were also tested in flexure and tension until the appearance of initial cracks, then placed in a water bath for given periods and re-tested so as to evaluate their ability to re-heal in humid conditions. The crack widths and possible mechanisms of healing have also been looked at. The results and their implications on the deployment of this material are presented in the following sections.

5.2 Mechanical Properties of naturally aged specimens

In order to assess the durability of the naturally aged material, specimens made from composition A (section 3.2.3) were prepared and placed in water at 20 °C representing typical curing conditions. The specimens were tested at different ages in flexure, according to the methodology detailed in section 3.4.3 so as to evaluate the change in mechanical properties with time. Figure 5-1 presents a comparison of the bending moment against curvature for specimens tested in flexure and aged 28, 307, 596, 896 and 1126 days. The load at first crack (Ffc), maximum load (Fmax), maximum curvature (Φmax) and flexural strength (σcf) of these specimens is summarised in Table 5-1. The flexural strength was calculated using equation 5.1, using l = 300 mm and d1 = d2 = 100 mm.

\[ \sigma_{cf} = \frac{F_{max} l}{d_1 d_2^2} \]  

(5.1)

Table 5-1: Flexural parameters of specimens of different ages

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Age (days)</th>
<th>Ffc (N)</th>
<th>Fmax (N)</th>
<th>Φmax (10^2 µm^-1)</th>
<th>σcf (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C2811</td>
<td>28</td>
<td>19148</td>
<td>23323</td>
<td>4.62</td>
<td>7.00</td>
</tr>
<tr>
<td>C2812</td>
<td>307</td>
<td>26091</td>
<td>31075</td>
<td>5.88</td>
<td>9.32</td>
</tr>
<tr>
<td>C2813</td>
<td>596</td>
<td>26396</td>
<td>33788</td>
<td>2.40</td>
<td>10.14</td>
</tr>
<tr>
<td>C2814</td>
<td>896</td>
<td>24131</td>
<td>24131</td>
<td>3.75</td>
<td>7.24</td>
</tr>
<tr>
<td>C2815</td>
<td>1126</td>
<td>23808</td>
<td>26576</td>
<td>3.26</td>
<td>7.97</td>
</tr>
<tr>
<td>C2816</td>
<td>1126</td>
<td>24069</td>
<td>29272</td>
<td>6.77</td>
<td>8.78</td>
</tr>
</tbody>
</table>
Figure 5-1: Bending moment-curvature graph of beam specimens tested at different ages, the black spots indicating the maximum curvature (Composition A – T1/P2)
Compared with plain concrete or a control specimen (without fibres), which exhibits stress relief after the formation of one macro-crack leading to the failure of the specimen (section 2.5.2), Figure 5-1 shows that most of the ECC specimens tested present an elastic region up to the onset of micro-cracking and as the bending moment continues to increase, further cracks are formed. The additional multiple fine cracks are formed as observed with the tensile specimens (section 4.2.2.3) revealing the pseudo-ductility of the material. The results are also in good agreement with Figure 2-14 presented by the Japanese Concrete Institute (2008).

As noted in section 4.2.3, some variation is to be expected with ECC material; this has been observed even between specimens from the same batch of material and is typical of a cured cementitious material. The behaviour of the ECC material observed is within the statistical variation seen within ECCs in general and the specific material used here. Whilst definitive conclusions on the effect of ageing on the mechanical performance could not be made, the results presented in Figure 5-1 indicate that the behaviour is maintained over some considerable time (more than three years).

As detailed in sections 2.5.3.7 and 2.5.3.8, ECC material exhibiting first crack at a lower load (compared with the maximum stress that the composite can withstand) would promote better pseudo-ductility. Introducing defects would limit the tensile failure strength of the cement matrix and hence maximum crack density could be achieved, increasing the strain capacity (Wang and Li, 2004). This is ideal for a matrix with a tendency to harden with time so promoting brittle behaviour. However, Figure 5-1 suggests firstly that the specimen with the higher load at first crack is not necessarily the oldest specimen and secondly, neither a lower nor a higher load at first crack will determine the best pseudo-ductile performance as the specimen with the highest ultimate curvature and a high maximum load is the one with an average load at first crack (specimen C2816). The results are promising for the application of this material where ductility is required and where ageing of the specimen could be associated with strength. Further testing is necessary to verify that the long-term performance is maintained over a longer period of time.

The strength of the material (cement matrix) increases with time as the matrix hardens (Rossi,
2013) and therefore could ultimately promote a high failure strength at first crack of the ECC material, hence the transfer of the load from the fibre to the matrix via the fibre-cement interface could be affected. This would promote a low crack density and hence reduce significantly the material pseudo-ductility. Hence, evaluating the strength of the cementitious matrix with time is very valuable for the determination of the ability of ECC material to form multiple cracks over its design life of 150 years.

As detailed in section 2.4.3, it is possible to make some estimate of the longevity of ECCs based on a prediction of the increasing strength of the cement matrix.

One of the conditions for the material to achieve multiple cracks under stress is linked with the presence of fibres above $V_{f,crit}$. On this basis, and following the approach of Naaman and Reinhardt (1996), equation 5.2 establishes the condition for the composite material to present a multiple cracking behaviour under stress, i.e. the ability of the fibres to take the load once cracks appear:

$$\sigma_{fu} \cdot V_f \geq \sigma_{mu} (1 - V_f) + \epsilon_{mu} \cdot E_f \cdot V_f$$  \hspace{1cm} (5.2)

Considering that $V_f$ is relatively small compared with $V_m$ and that $E_f \approx 3E_m$, then equation 5.2 becomes:

$$\sigma_{fu} \cdot V_f \geq \sigma_{mu} (1 - V_f)$$  \hspace{1cm} (5.3)

Equation 5.3 requires the determination of the matrix strength at 150 years in order to verify whether pseudo-ductility is maintained with time. Data presented by Shariq et al. (2008) showing the compressive strength of cement mortar with time enables the plotting of Figure 5-2. Figure 5-2 illustrates the evolution of the cementitious compressive strength with time (depending on the water/cement ratio) and eventually reaching a plateau.

The best fit for the data obtained from Shariq et al. (2008) enables the determination of an equation estimating the compressive strength $\sigma_{cs}$ with time $t$:  

175
\[ \sigma_{cs}(t) = 7.2337 \ln(t) + 19.414 \]  

(5.4)

Using the time \( t \) of 54750 days (equivalent to 150 years) in equation 5.4, the compressive strength of the cement mortar is estimated to be 98.3 MPa (the highest value, based on the model). Eren (2002) covering the strength development of Portland cement concretes estimated the ultimate compressive strength of 30 \% PFA/ 70\% PC blend at 85 MPa. Shariq et al. (2008) demonstrated that the compressive strengths of concrete and cement mortars have similar values (= 53 MPa at 180 days), hence the value of 98.3 MPa could be considered as the upper limit of the ultimate compressive strength of cement mortar.

Neville (2000) suggests that an expression used in the British Code of Practice (BS 8007: 1987) gives a clear link between the tensile and compressive strength of the cementitious (concrete) material:

\[ \sigma_{ts} = 0.12(\sigma_{cs})^{0.7} \]  

(5.5)

where \( \sigma_{ts} \) is the direct tensile strength of the cementitious material.
Using a compressive strength $\sigma_{cs}$ of 98.3 MPa at 150 years age, the direct tensile strength is estimated at 2.98 MPa at 150 years.

Using $\sigma_{tu}$ equal to 1560 MPa, $V_f$ as 2 % vol., $\sigma_{mu}$ as 2.98 MPa then equation 5.3:

$\sigma_{fu} V_f \geq \sigma_{mu} (1 - V_f)$ gives 31.2 MPa $\geq$ 2.92 MPa at 150 years.

Hence, assuming that the fibre properties remain intact, the increase in the cement matrix strength over time is not an obstacle to the pseudo-ductility of ECC materials.

The second condition presented in section 2.4.3 is the following:

$$\sigma_{pc} \geq \sigma_{cc}$$

(5.6)

where:

$$\sigma_{cc} = \sigma_{mu} (1 - V_f) + \alpha \tau_o V_f \frac{L_f}{d_f}$$

(5.7)

$$\sigma_{pc} = \lambda \tau_o \frac{L_f}{d_f} V_f$$

(5.8)

This leads to:

$$V_{f, crit} = \frac{\sigma_{mu} d_f \frac{1}{L_f \lambda - \alpha}}{\tau_o}$$

(5.9)

Considering $\lambda - \alpha = \eta_0$, then using $\sigma_{mu} = 2.92$ MPa, $\tau_0 = 1.62$ MPa, $d_f = 40 \mu m$, $L_f = 8$ mm and $\eta_0 = 0.51$, 0.37 and 0.31 for specimens C6523, C6543 and C6544C respectively, then $V_{f, crit}$ is estimated at 0.018. The result illustrates the suitability of $V_f = 0.02$, as used in current mixes, for maintaining the pseudo-ductility.

5.3 Potential effect of ageing on the fibre-cement interface

5.3.1 Introduction

As discussed in section 2.5.3.5, the fibres have been specifically tailored with a surface coating to offer the appropriate pull-out resistance when incorporated in a cementitious matrix. The
coating is essential to the maintenance of the pseudo-ductile behaviour of ECC material over time. Hence, the long-term durability of the material is dependent on the integrity of the coating and changes in the strength of any interfacial bonding over time. In this section, three areas giving information about the fibre-cement interface are investigated:

- Stability of the fibre surface coating
- Chemistry of the fibre-cement interface
- Single-fibre pull-out testing

Within each section, a brief outline of expected outcomes is given, together with results and some discussion of the impact on durability.

### 5.3.2 Stability of the fibre coating

#### 5.3.2.1 Introduction

As outlined in section 3.6.2, time-of-flight secondary-ion-mass-spectrometry (ToF-SIMS) has been used to characterise the surface of the fibres and the tailored coating applied to them. Further, the stability of the coating after long-term exposure to (tap) water has been evaluated.

The supplier has stated that the fibres are coated with a combination of seven different hydrocarbon molecules, present at 1.2 wt. %. Due to confidentiality agreements, it is not possible to specify the constituent molecules of the coating but, in general, the composition of this coating may be described as:

- 75 wt % one short chain hydrocarbon, which cannot bond strongly with cement
- 24.9 wt % two long chain hydrocarbon molecules which can bond strongly with cement
- Small quantities (maximum content 0.1 wt %) of halogenated hydrocarbons (Br + Cl) as well as molecules containing N + S, adding up to 100 %.

The latter molecules in particular, whilst present in small quantities, can be identified using ToF-SIMS. Br and Cl for example, have a sequence of naturally occurring isotopes that are present in very specific ratios; which can be used to provide a “finger-print” for the chemistry. Similarly, the presence of Nitrogen is helpful because it gives (unusual) even numbered peaks, which are relatively easy to assign. Hence, these peaks will form the basis for the comparison made. Table 5-2 presents the key elements to be searched and their respective isotopes.
In order to assess the durability of the coating, samples of the coated fibre were kept in water for nearly three years. The surface chemistry of these fibres was compared with that of the as-received (dry) oiled fibres. The coating could be present at, at most, one monolayer and hence could be detected using the SIMS technique along with the core of the PVA fibre. The results of the analyses are presented in graphs where the relative peak intensity is plotted against the atomic mass. The relative intensity is the normalised intensity of each peak: each peak intensity is divided by the sum of the intensities.

The results are also compared with the positive and negative spectra of the PVA molecule found in the literature (Surface Spectra, 1999) in order to confirm the presence of a coating (Figure 5-3).

5.3.2.2 Results and discussion

Figures 5-4 and 5-5 present the analysis of the fibres (as received) kept in air and Figures 5-6 and 5-7, the analysis of the aged fibres kept in water at 20 °C for about three years, where the relative intensity is plotted against the atomic mass. The depth of analysis of ToF-SIMS is in the order of 1-2 atomic layer(s), hence if the coating is present as a complete monolayer, it is unlikely that the core of the PVA fibre will be seen. On the other hand, a more patchy coating will mean that the fibre will be quite obvious. The PVA fibre has been characterised by ToF-SIMS and a reference spectrum of a PVA molecule is available in the literature for comparison (Figure 5-3; Surface Spectra, 1999); it should be noted that the highest atomic mass presented in this reference is 300 m/z (u).

In order to assign the peaks accurately, the width of the hydrogen on the spectra has been checked. This is a standard process to indicate confidence in the resolution of the peaks when using ToF-SIMS. The maximum acceptable width of the hydrogen peak (which, due to the

<table>
<thead>
<tr>
<th>Name</th>
<th>Isotopes Mass (Abundance %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrogen (N)</td>
<td>14.003 (99.63) 15.000 (0.37)</td>
</tr>
<tr>
<td>Chlorine (Cl)</td>
<td>34.969 (75.77) 36.966 (24.23)</td>
</tr>
<tr>
<td>Sulphur (S)</td>
<td>31.972 (95.02) 32.971 (0.75) 33.968 (4.21) 35.967 (0.020)</td>
</tr>
<tr>
<td>Bromine (Br)</td>
<td>78.918 (50.69) 80.916 (49.31)</td>
</tr>
</tbody>
</table>
nature of the data collected is determined in terms of time, whilst m/z (u) gives position) for confidence in the subsequent assignment of peaks, is a width of 1.0 – 1.1 ns: in the current work, 0.84 ns was achieved. The spectra have also been calibrated using peaks corresponding to a number of elements (including H and C).
Figure 5-3: (a) Negative ion spectrum and (b) Positive ion spectrum of a standard poly(vinyl)alcohol molecule (SurfaceSpectra, 1999)
Figures 5-4 and 5-5 show, as expected, fragments of molecules of small chains and then long chains as the atomic mass increases. The presence of peaks beyond 300 m/z (u) confirms the existence of a coating on the surface of the fibres. The existence of repeated units, mainly visible at higher atomic masses, are due to the presence of long carbon chains constituting the coating and/or the PVA fibre core. Comparing Figure 5-4 with the negative spectra of the PVA molecule presented in Figure 5-3, similar peaks are visible at lower atomic masses. The peak corresponding to CH ion at 13 m/z (u) is greater in intensity than in Figure 5-3 and this is likely to be due to the presence of the coating. Hence, the presence of peaks typical of PVA fibres and additional peaks particularly at higher atomic mass suggest that both the fibre core and the coating are detected, which confirms that the coating is present as a very thin layer and at most one monolayer (probably one atomic layer) as expected, or as a patchy coating, which also enables the detection of the PVA core.

Figure 5-4b shows the presence of Chlorine, which as indicated has two isotopes, one at 34.967 m/z (u) and another at 36.966 m/z (u), the relative peak intensity of these being 0.00016 and 0.0000513 respectively (i.e. a ratio of 3:1, which corresponds well with the abundance of these isotopes in nature which is 75.77 % for Cl$^{35}$ and 24.23 % for Cl$^{37}$). Sulphur, another element present in the fibre surface coating, was also detected at 31.972 m/z (u), this isotope being present in nature at 95.02 %.

Figure 5-5 presents the positive ions SIMS spectrum of the fibres kept in air. Comparing the positive spectrum with the one presented in Figure 5-3, it appears that similar peaks are present at lower atomic mass (for example at 15 and 19 m/z (u)). Additional peaks such as the ones at 30 and 46 m/z (u) that are not usually present in the standard spectrum (Figure 5-3), could be a part of the coating or, potentially contamination. Also, peaks containing carbon such as the one corresponding to C$_2$H$_5$+ are larger, which probably also comes from the coating. The results also show the presence of sodium as an impurity and the presence of fragments of long carbon chains at higher atomic mass. The (N-CH$_3$)$^+$ ion present in the coating has been detected at 29 m/z (u). The analysis reveals the presence of a coating and peaks typical of the PVA molecule.
Figure 5-4: Negative ions SIMS spectrum for the PVA fibre kept dry: (a) up to 50 m/z (u), (b) from 50 to 100 m/z (u), (c) from 100 to 300 m/z (u) and (d) from 300 to 750 m/z (u)
Figure 5-5: Positive ions SIMS spectrum for the PVA fibre kept dry: (a) up to 50 m/z (u), (b) from 50 to 100 m/z (u), (c) from 100 to 300 m/z (u) and (d) from 300 to 750 m/z (u)
Figure 5-6: Negative ions SIMS spectrum for the PVA fibre kept in water for nearly 3 years: (a) up to 50 m/z (u), (b) from 50 to 100 m/z (u), (c) from 100 to 300 m/z (u) and (d) from 300 to 750 m/z (u)
Figure 5-7: Positive ions SIMS spectrum for the PVA fibre kept in water for nearly 3 years: (a) up to 50 m/z (u), (b) from 50 to 100 m/z (u), (c) from 100 to 300 m/z (u) and (d) from 300 to 750 m/z (u)
The fibres kept in water for nearly three years were dried and then analysed in the same manner as the “as supplied” fibres. Figures 5-6 and 5-7 show, similarly to the dry “as supplied” fibres, the existence of fragments of long-chain hydro-carbons, confirmed by the existence of repeated units from lower to higher atomic masses (and the (N-CH₃)⁺ ion, part of the surface coating is also present), but perhaps more importantly, Figure 5-6b indicates a similar relation between the isotopes of chlorine as observed in the “as supplied” dry fibres. The chlorine isotopes are present in a ratio of 3:1, the relative peak intensities being 0.000160 and 0.0000497 respectively. This, with sulphur containing ions, show that the coating is still present although the fibres have spent nearly three years in water. Whilst relative intensities cannot be treated as absolute values and although some peaks between 40 and 45 m/z (u) tend to be considerably reduced, it is interesting to note that the relative intensities are similar for the aged fibres as for the “as supplied” ones, and the ratio of abundance is preserved. This suggests that the coating is still present and intact.

5.3.2.3 Discussion
SIMS analysis of the fibres, comparing the “as supplied” dry fibres with the fibres kept in water for nearly three years suggests that the coating remains intact over time. The finding is promising, and is suggestive that pseudo-ductility of the material will be maintained over time. However, care should be taken when interpreting the results, as cement solution, which could also deteriorate the fibres is different to simple water.

The surface analysis and the full modelling of the chemical interaction of the fibre with the matrix and the role of the (tailored) surface coating could form the basis of a research project in its own right and such an in-depth analysis is beyond the scope of the current work. This limited study has shown that the coating is present and intact after long-term immersion in water, but care should be taken when extrapolating these results to a cement solution and/or behaviour over increasing lengths of time.

5.3.3 Chemistry of the fibre-cement interface

5.3.3.1 Introduction
The long-term durability of the ECC material is also associated with the ageing process at the fibre/cement interface, as changes here will affect the pseudo-ductile behaviour of the
material over time. Hence, the addition of a coating on the surface of the fibres. As detailed in section 2.5.3.5, the PVA fibre-cement interface is characterised by the presence of a layer of Ca(OH)$_2$ around the fibre showing that the PVA fibre is likely to interact with the ions from the cementitious matrix, this is especially relevant as the fibre coating may be present in a patchy way. The formation of this strong bond due to the presence of hydroxyl groups on the PVA backbone in its molecular chain could prevent the fibres from pulling-out from the cementitious matrix when the ECC material is under stress: the fibres could break, which could reduce the pseudo-ductility of the material. Information about the type of bonding between the two materials and changes occurring will therefore give an indication of the ageing process at the interface and hence the long-term ability of the fibre to pull-out in the cement matrix under stress.

Figure 5-8 shows a fracture surface of an ECC material exhibiting fibres sticking out from a cementitious matrix. The fibres exhibit cementitious material on their surface, revealing a certain interaction between the fibre and the cement, hence demonstrating the possible existence of a (chemical) bond between the fibre and the cement.

![SEM image of fibres sticking out of the cementitious matrix at the fracture surface of a specimen](image)

**Figure 5-8: SEM image of fibres sticking out of the cementitious matrix at the fracture surface of a specimen**

An attempt has been made to characterise the chemistry of the fibre-cement interface and
the chemical bonds present. This is carried out by comparing the XPS spectrum of the surface of the “as supplied” fibres with the surface spectrum analysis of the pulled-out fibre previously embedded in the cementitious matrix, where cement is usually intimately attached (Figure 5-8). The “as supplied” and pulled-out fibres are placed on a support and analysed using the X-ray photoelectron spectroscopy (XPS) characterisation technique as presented in section 3.6.3.

5.3.3.2 Results
The results are compared with a reference spectrum of the PVA molecule (Figure 5-9). A survey spectrum for each fibre type is presented, fibre type T1 “as supplied” in Figure 5-10 and a cement coated fibre in Figure 5-11. A high resolution spectra of the carbon, oxygen and calcium peaks (for both fibres) are presented in Figure 5-12, complete with peak fitting assignments.

Figure 5-10 presenting the XPS survey spectrum of the “as supplied” fibres shows the presence of two main peaks: a peak of carbon at 285 eV and a peak of oxygen relatively lower in intensity, as expected. Auger peaks are also visible, these are usually broader peaks compared with the sharper XPS peaks. However, the standard (Figure 5-9) shows that the polyvinylalcohol (PVA) molecule spectrum is usually characterised by a higher intensity oxygen peak in comparison with the carbon peak. Hence, this analysis supports the presence of a coating with a high carbon content as suggested by the supplier in section 5.3.2.1.

Figure 5-11 presents the XPS survey spectrum of the pulled-out fibre recovered from fractured surfaces of ECC test coupons. However, a lower carbon/oxygen ratio is detected compared with the ratio obtained for the fibres on their own, some of the oxygen detected could come from the interface or the cement. The results suggest that the presence of calcium may come from the cement, and possibly the interface, as the literature mentions the existence of a strong interaction between the cement and the fibres characterised by a Ca(OH)\textsubscript{2} layer around the PVA molecule (section 2.5.3.5), forming a chemical bond between cement and fibre (Li et al., 2001).

The peak fitting of the elements such as Carbon (C1s), Oxygen (O1s) and Calcium (Ca2p) would give valuable information as to the chemical bonding. As detailed in section 3.6.3, the chemical
shifts brought about by the various bonds between two different elements enables the determination of the chemical species that are bound to the element of interest. Hence, the high resolution spectra of key elements enable peak fitting to be carried out, which can be used to determine the nature of the bonding taking place and hence the chemical species present. Figure 5-12 presents such spectra for carbon, oxygen and calcium. From these, it can be seen that there is a change in the kinds of bonds occurring, particularly in terms of carbon bonds (some are due to the presence of calcium carbonate, and some due to bonding between the fibre and the cement).

Figure 5-9: XPS (a) survey spectrum a standard poly(vinyl)alcohol (PVA) molecule and (b) spectrum of its carbon peak fitting (Beamson and Briggs, 1992)
Figure 5-9: XPS spectrum of type T1 fibre as-received
Figure 5-10: XPS spectrum of pulled-out fibre (type T1 REC) from specimen C2742
Figure 5-12a presents the detailed XPS spectrum analysis of the carbon peak and compares it to the literature, the peaks observed correspond to C-C, C-H (285 eV) and C-OH (286.47 eV), which are typical of a PVA molecule. In addition, another peak is present at higher binding energy and in small proportion, which corresponds to C=O, the latter may be due to the coating presenting this type of bond (or could be a residue of the polyvinylacetate used to make PVA). Comparing the results with Figure 5-12b, similar bonds are present, but in addition peaks relative to HO-C=O and O-C=O(O) (carbonate), which means that the carbon from the fibre could interact with its environment. Knowing that the C=O would probably come from the coating, it then appears that this specific bond may form an ionic bond with the oxygen ions (O²⁻) dissolved from the cementitious material, which dissolves once cement is in contact with water or from the hydroxyl group of the fibre. The carbonate group, not present in the fibre coating, could either come from cement or the C=O bound twice to oxygen ions from cement.

Figure 5-12c presenting the oxygen peak fitting scan shows a “chemical” bond C-OH related to the PVA fibre on its own, however Figure 5-12d reveals additional “chemical” bonds for the fibre coated with cement. These additional peaks correspond to carbonate, and also C=O. These confirm the finding made previously. Figure 5-12e illustrates the chemical shift of the oxygen from a fibre covered with cement to a fibre on its own. The fibre covered with cement exhibits a broader peak due to the high number of bonds and the chemical shift relative to the “chemical” bonds.

Figure 5-12f presents the calcium peak from the fibre covered with a cementitious matrix. The calcium is present at 346.6 eV, hence the chemical shift clearly indicates its interaction with the other chemical species present. The peaks most likely correspond to CaCO₃ and CaAl₂O₃, both being present in Portland cement. The chemical shift might be due to the chemistry of cement, in particular the various ionic bonds in play shifting the balance of electrons.

In summary, the study confirms the interaction of the fibre with its environment and enables the determination of possible species constituting the fibre-cement interface.
Figure 5.11: XPS spectra (narrow scan) from the as-received fibres of (a) carbon, (c) oxygen and from the fibre pulled-out from a cementitious matrix of (b) carbon, (d) oxygen and (e) calcium and (d) comparison of oxygen from as-received fibre and fibre pulled-out from a cementitious matrix.

5.3.3.3 Discussion

An attempt has been made to analyse the chemistry of the fibre-cement interface by analysing the fibres pulled-out from the cementitious matrix, as cementitious material was sticking on the fibres’ surface. The analysis showed the presence of elements belonging to the fibres and also cement such as calcium, this conforms to the possible bond made of calcium and elements belonging to the fibres such as oxygen. As noted in the previous section, this work has been carried out in order to answer very specific questions about the level of bonding.
observed and hence the durability of the coating and the stability of the bonding between the fibre and matrix. This has raised further questions, but it is beyond the scope of the current work to explore further at this time and more data is required to make a conclusion on the fibre-cement bond.

5.3.4 Single fibre pull-out (SFPO) testing

5.3.4.1 Introduction
As discussed in section 2.5.3.5, single fibre pull-out testing of fibres of different ages from a cementitious matrix provides interfacial properties that help evaluate the fibre-cement bond properties with time and hence the mechanical performance of the ECC material in tension over time. The behaviour observed in cement-PVA fibre testing, a number of different pull-out tests and their meaning in terms of the interaction are discussed.

Multiple cracking is dependent on the ability of the fibres to appropriately slip in the matrix when a crack forms. In turn, this ability to slip is dependent on the bonding between the fibre and the matrix and as discussed in section 2.5.3.5, this can initially be characterised by single fibre pull-out (SFPO) testing. Three physical characteristics are quantified:

- $\tau_d$: interfacial mean shear strength, characterises the initial debonding mechanism of the fibre from the cementitious matrix as it takes into consideration the maximum load that the fibre can withstand before debonding.
- $G_d$: chemical debonding energy, characterising the second stage of the pull-out testing curve, and is closely linked to the sudden load drop once the fibre/cement chemical bond is broken.
- $\tau_0$: frictional bond strength, characterises the last stage of the single fibre pull-out testing curve, the load per square meter when the fibre is dislodged from the cementitious matrix.

In addition, $\beta$, a geometric parameter characterising the slippage regime, is determined where:

- $\beta < 0$ indicates a slip-softening regime
- $\beta = 0$ characterises a constant friction slippage regime
- $\beta > 0$ shows a slip-hardening regime
SFPO data for specimens aged 28 days have been presented in Chapter 4. Here, this data is compared for a range of ages: 7 days (22 tests), 28 days (21 tests), 3 months (20 tests) and 5 months (20 tests), as outlined in section 3.4.4 which also details the sample preparation. This data gives an indication of the evolution of the interfacial parameters with time and could predict whether ECCs will be able to maintain pseudo-ductility under tensile stress in the long-term.

5.3.4.2 Results

Typical single fibre pull-out profiles have been presented in section 4.4.1 for specimens aged 28 days. Figure 5-13 presents similar responses for fibres pulled-out from a cementitious matrix, where the samples are aged 7 days, 28 days and 3 months and also shows the aspect of the fibre at the end of testing. The graphs in Figure 5-13 show, in the first stage of the testing, an increase of the load up to the first crack followed by a sudden drop of the load. However, three different cases are evident in the second stage of the pull-out testing:

- Figure 5-13a shows, for a sample aged 7 days that the load decreases constantly with the displacement exhibiting a constant friction regime towards the end of testing. This result corresponds to a fibre kept intact.
- Figure 5-13b for a sample aged 28 days, reveals a decrease of the load with the displacement and this decreases with time. This result also corresponds to a fibre kept intact.
- Figure 5-13c, corresponding to a sample aged 3 months, shows a load increase with the displacement in the second stage of the testing. This result is associated with a slip-hardening regime and corresponds to a damaged fibre. A slip-hardening regime would be associated with a stronger fibre-cement bond and according to the literature, this regime is more likely to be visible when the fibres are less rigid than the matrix, as with PVA fibres in a cementitious matrix. The results again show the intimate fibre-cement bond and also that the response of an ECC material to a tensile stress depends on this bond strength.

The interfacial mean shear strength $\tau_d$, the chemical debonding energy $G_d$ and the frictional bond strength $\tau_0$ are plotted against time and are presented in Figure 5-14. Table 5-3 summarises the results obtained from a number of different specimens tested for each age.
Figure 5-12: Single fibre pull-out load-displacement responses of selected samples (with the corresponding SEM image of the sample) aged (a) 7 days, (b) 28 days and (c) 3 months.
Figure 5-13: Interfacial parameters calculated through single fibre pull-out testing for samples aged 7 days, 28 days, 3 months and 5 months: (a) Interfacial mean shear strength, (b) Chemical debonding energy, c) Frictional bond strength
Table 5-3: Interfacial bond properties of specimens aged 7 days (22 tests), 28 days (21 tests), 3 months (20 tests) and 5 months (20 tests)

<table>
<thead>
<tr>
<th>Time (days)</th>
<th>$\tau_d$ (MPa)</th>
<th>Std dev.</th>
<th>$G_d$ (J.m$^{-2}$)</th>
<th>Std dev.</th>
<th>$\tau_0$ (MPa)</th>
<th>Std dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>2.8</td>
<td>1.8</td>
<td>1.8</td>
<td>1.0</td>
<td>1.3</td>
<td>1.0</td>
</tr>
<tr>
<td>28</td>
<td>3.6</td>
<td>1.7</td>
<td>3.0</td>
<td>1.4</td>
<td>1.5</td>
<td>1.0</td>
</tr>
<tr>
<td>91</td>
<td>3.3</td>
<td>2.3</td>
<td>2.9</td>
<td>1.8</td>
<td>1.5</td>
<td>1.4</td>
</tr>
<tr>
<td>152</td>
<td>3.7</td>
<td>2.5</td>
<td>2.2</td>
<td>1.3</td>
<td>1.9</td>
<td>1.4</td>
</tr>
</tbody>
</table>

Figure 5-14 shows that $\tau_d$, $G_d$, $\tau_0$ remain essentially constant with time ($\tau_0$ is within the range specified in the literature for pseudo-ductile behaviour, from 1 to 2 MPa), demonstrating that the fibres are still able to take a load even when aged and the fibre-cement interface is able to offer the appropriate resistance to the fibre pull-out, which is quite promising for the long-term durability of the ECC material, although care should be taken as 5 months is far from a duration of more than 100 years. It might be argued that there is a slight change in $G_d$ (an increase and then a decrease), but the values measured are within the range specified in the literature for oiled fibres tailored for ECC (0.5 to 9.5 J m$^{-2}$). The values of $\tau_0$ show that the fibres would have the ability to exhibit the same behaviour in the slippage regime (if the slippage regime is dependent on its initial frictional bond strength). It should be noted that pseudo-ductility of ECC material could be affected if $G_d$ follows this trend with time. At this stage it is unclear whether $G_d$ will reach a plateau or not with time, however the 5 months data reveal that the results observed are within the variation of the ECC material (error bars), hence this effect can be neglected.

Table 5-4 presents the number of pull-out tests for each slippage regime mode. Specimens aged 7 and 28 days show a similar number of samples with slip-hardening and slip-softening behaviour. However with time, there seems to be an equal number of specimens presenting all three behaviours in the slippage regime. A slip-hardening regime would be highly beneficial within the ECC material, showing that the fibre is able to withstand a higher load. In addition, ECC is more likely to exhibit a slip-hardening regime as the polymeric fibres, which are less hard than the surrounding matrix, can damage and a jamming effect can take place within the matrix leading to an increase in load resisting fibre pull-out (Redon et al., 2001). Therefore, as the number of slip-hardening regimes is decreasing with time meaning a larger number of constant friction and slip-softening regimes, it could be believed that the stronger fibre/cement interaction with time could promote harder fibre systems and therefore less
slip-hardening regimes. However, the data obtained at 5 months shows that the slip-hardening regime is not decreasing with time, which is promising for the long-term durability of ECC material.

Table 5-4: Number of each slippage regime mode type per pull-out test age

<table>
<thead>
<tr>
<th></th>
<th>7 days</th>
<th>28 days</th>
<th>3 months</th>
<th>5 months</th>
</tr>
</thead>
<tbody>
<tr>
<td>β &lt; 0</td>
<td>11</td>
<td>9</td>
<td>7</td>
<td>7</td>
</tr>
<tr>
<td>β = 0</td>
<td>2</td>
<td>2</td>
<td>7</td>
<td>4</td>
</tr>
<tr>
<td>β &gt; 0</td>
<td>9</td>
<td>10</td>
<td>6</td>
<td>9</td>
</tr>
</tbody>
</table>

5.3.4.3 Summary
The single fibre pull-out (SFPO) tests revealed valuable information over a period of 5 months. The values measured are within the range of those specified in the literature to obtain a pseudo-ductile behaviour as detailed with the 28 days data in section 4.4. The values remain nearly constant with time and within the variation observed within the material demonstrating a maintaining of the pseudo-ductile behaviour over 5 months, and hence probably over a longer period of time. However, there is too much scatter in the data to make definite conclusions within the measured period. Overall, it can be concluded that the fibre-cement interface is relatively stable and is likely to remain so for a significant period of time.

5.4 Self-healing
5.4.1 Introduction
Cracks appear routinely during the life of cementitious materials, due to shrinkage or various imposed operational stresses, or a combination of these. In section 2.6.5, the ability of the material to self-heal after the formation of cracks was introduced; this is called autogenous healing. In this section, the progress made on the autogenous healing ability of ECC material is presented. The ECC material (using specimens made with fibre type T1 and process P2) is tested in flexure and in tension, the crack formation and widths are investigated and the possible mechanism of healing is presented.

5.4.2 Flexural performance
Figure 5-15 presents the bending moment against the curvature of a specimen initially tested in flexure at 43 days until the appearance of initial cracks, then returned to a water bath (still at 20 °C) before being re-tested at 95 days. The initial flexure testing experiments undertaken
reveal the possibility of the ECC material to exhibit ductility with time even when subject to a prior stress causing the formation of cracks in the specimen (Figure 5-15). The first observation is that the material when re-tested exhibits an increased load at first crack compared to when tested initially, this could be due to ageing of the specimen with time. Then at lower loads, the re-tested specimen seems to have a similar Young’s modulus to initially; it follows the same curve, which could suggest the healing of the initial cracks. However at higher loads, once reaching a bending moment of 0.26 MN.mm, the ECC material exhibits a sudden change in Young’s modulus in the elastic region of the curve, this suggests the sudden opening of existing cracks, hence revealing a partial healing of the existing cracks. It appears possible that the increase of performance when re-loaded is due to the (partial) healing of cracks in the material. Furthermore, it has been observed that when re-testing the specimen, new cracks appear followed by the opening of previously opened cracks, reinforcing the hypothesis of the partial healing effect of the ECC material. However, complementary tests such as the evaluation of the crack formation of the specimens being re-tested would be beneficial; this could give an indication of the degree of healing ability of the ECC material.
Figure 5-14: Pre-loading and re-loading bending moment – curvature of ECC specimen (Fibre T1/Process P2)
5.4.3 Tensile performance

5.4.3.1 Introduction

The experiments conducted in flexure have proved that ECCs have the ability to at least partially self-heal. Following these initial results, specimens kept in water at 20 °C were also tested in tension initially, and then returned to the water bath (still at 20 °C) before being re-tested as detailed in section 3.4.2.4. Two effects on subsequent performance have been evaluated:

- The initial pre-cracking magnitude at the first test
- The curing duration after the initial test and before the second test

The testing programme conducted will enable a better understanding of the limitations of the ECC material on its ability to withstand further load when initially damaged.

In this section, Figures 5-16 and 5-17 present the tensile stress as a function of strain corresponding to the cross-head displacement of the machine (and to the “real” strain defined as the strain after the onset of cracking). Figure 5-18 shows the tensile stress as a function of the “real” strain measured with the use of an LVDT and strain-gauges as detailed in section 3.4.2.4.

5.4.3.2 Effect of initial pre-cracking magnitude on subsequent performance

Figure 5-16 presents the results of specimens kept in water, initially tested at 28 days up to a displacement of 0.3 mm (C7124-1) and 0.5 mm (C7123-1) as measured by the LVDT, then returned in the water bath (still at 20 °C) before being re-tested after a further 28 days cure. The specimen initially tested up to a displacement of 0.3 mm can withstand a stress of just above 3.5 MPa and then when re-tested, the tensile stress at the end of the elastic region seems to be identical. In addition, the re-tested specimen seems to exhibit a strain of 1.8 % after the first crack, which is very close to the strain seen in section 4.2.3 for a typical non-damaged ECC specimen. On the other hand, the specimen initially tested up to a displacement of 0.5 mm, when re-tested, exhibits its initial crack at a similar stress to when initially tested, around 3.5 MPa. However, the strain after first crack reaches only 0.58 %. This might be due to the fact that the latter specimen has been initially tested up to a higher displacement (0.5 mm) and hence is more damaged. Further testing is required to confirm this hypothesis. Also, interestingly, for the application of the material in civil engineering, it was observed during
testing that the specimen would rather form new cracks than opening existing ones, especially at lower strains which will be verified further in this section.

### 5.4.3.3 Effect of curing duration after the initial test and before the second test

Figure 5-17 details the results of specimens initially tested in tension at 28 days to similar displacements (of 0.5 mm as recorded by the LVDT), then returned to the water bath (still at 20 °C) for different durations: 14 days (C7542), 28 days (C7541) and 42 days (C7540), before being re-tested.

Initial observations show that the specimen re-tested at 14 days is able to withstand a similar or higher load than when being initially tested, whilst exhibiting further multiple cracks (C7542). Similar observations are made for the specimens re-tested after being cured in water for 28 days and 42 days (C7541 and C7540 respectively). It is interesting to note the difference in strain after first crack for these two specimens, although, given the variability in the material and the small data set, definite conclusions cannot be made at this stage. However, the results demonstrate that the specimen is capable of performing well when initially damaged, and that the subsequent performance is rather a material property than depending on the duration of curing. As expected, each specimen is unique and represents a specific arrangement of the fibres and the constituents in the cementitious matrix.

The curing time seems to have an insignificant effect on further mechanical performance of the ECC material (maximum stress and ultimate strain) when being re-tested. A longer curing time in water does not specially lead to a better performance in tension when the material is re-tested. Further investigation is required to determine the possible factors influencing the self-healing ability of the ECC material (such as crack widths, water characteristics...) along with possible healing mechanisms and products. In addition, further analysis of the displacement associated with the specimen only (using strain gages and LVDT) would enable an accurate estimation of the Young’s modulus values of the material pre-tested and re-tested and would confirm the initial observations.
Figure 5-15: Tensile stress-strain relationships. Pre-loading up to (a) 0.3 mm specimen C7124-1 and (b) up to 0.5 mm specimen C7123-1; and re-loading specimens C7124-2 and C7123-2 after 28 days cure - Specimens made with fibre type T1 and process P2
Figure 5-16: Tensile stress-strain relationships. Specimens pre-tested up to 0.5 mm (recorded on the LVDT) at 28 days before being re-tested after curing in water at 20 °C and after (a) 14 days (C7542), (b) 28 days (C7541) and (c) 42 days (C7540) – Specimens made with fibre type T1 and process P2.
5.4.3.4 Effect of time on the stiffness of ECC material

As the trend observed with the previous experiment was not conclusive, which is especially relevant due to the variability observed within specimens made from the same batch, it was decided to test one specimen successively and to follow its behaviour with time after similar curing periods. This will most closely represent reality. The effect of initial damage and also time is evaluated on further mechanical performance of the same specimen, with particular attention to the evolution of the initial Young’s modulus and stiffness.

Figure 5-18 presents the tensile stress associated with the strain, the values reported being associated with the displacement of the specimen only (as opposed to the cross-head displacement of the machine). Table 5-5 presents the evolution of the Young’s modulus for the specimens tested, E represents the initial Young’s modulus of the material before the opening of the existing cracks (at lower load) whilst \( E^* \) is the Young’s modulus of the specimen at higher load once some of the existing cracks re-open. Hence, \( E^* \) is lower in value than E. The specimen when tested initially exhibits an initial high tangent Young’s modulus (C7125-1) equal to 13.2 GPa and similarly when being re-tested (C7125-2) up to a load corresponding to a specific stress (1.5 MPa), however, the stiffness then reduces to a value of 4.6 MPa (Table 5-5). A similar Young’s modulus demonstrates that the specimen has healed, however, when the load reaches a specific value, some of the old cracks start to open again with increased strain before complete opening of the crack, hence demonstrating the partial healing of the ECC material. This trend continues with further cycles of testing until the specimen is tested to failure (9th test) and exhibit a stiffness of 1 GPa (\( E^* \)) having had an initial Young’s modulus of 13.2 GPa (E). These findings are very promising for the use of this material in civil engineering applications showing that the ECC material is able to exhibit similar properties from before being damaged and to having been damaged up to 8 times with a multiple-cracking behaviour under tensile stress as observed in the final testing.

Comparing the experimental values obtained here with the theoretical values using the ACK theory (section 4.5.2), it appears that the value of 13.2 GPa is in line with the prediction suggesting that a value of about 13 GPa is correct. The measured strain at first crack for specimen C7125 had an average value of 0.0274 %, equal to a strain \( \varepsilon_{\text{mu}} = 0.00027 \), which is very close to the value predicted by the ACK equation considering fibre orientation (values
from $\varepsilon_{mu} = 0.00022$ to $0.00030$ for $\eta$ values from 0.31 to 0.51).

Table 5-5: Initial Young’s modulus before the opening of existing cracks ($E$) and after the opening of existing cracks ($E^*$) – N/A meaning that the data is not available

<table>
<thead>
<tr>
<th>Test No</th>
<th>Months</th>
<th>Displacement (mm)</th>
<th>$E$ (GPa)</th>
<th>$E^*$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>0.2</td>
<td>13.2</td>
<td>13.2</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>0.4</td>
<td>13.2</td>
<td>4.6</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>0.6</td>
<td>13.2</td>
<td>N/A</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>0.8</td>
<td>13.2</td>
<td>1.8</td>
</tr>
<tr>
<td>5</td>
<td>7</td>
<td>0.6</td>
<td>13.2</td>
<td>1.4</td>
</tr>
<tr>
<td>6</td>
<td>8</td>
<td>0.6</td>
<td>13.2</td>
<td>1.3</td>
</tr>
<tr>
<td>7</td>
<td>9</td>
<td>0.6</td>
<td>13.2</td>
<td>1.5</td>
</tr>
<tr>
<td>8</td>
<td>10</td>
<td>0.6</td>
<td>13.2</td>
<td>1</td>
</tr>
<tr>
<td>9</td>
<td>11</td>
<td>up to failure</td>
<td>13.2</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 5-19 shows the specimen C7125 after being tested to complete failure and the evolution of the ECC Young’s modulus as a function of time and previous damage displacement.

The specimen exhibits cracks in the whole area of the gauge length where the dispersion of cracks seems excellent (Figure 5-19a). Figure 5-19b shows that an increase in displacement damage results in a decrease of the ECC material Young’s modulus ($E^*$). The important curing duration (3 months) between Tests No 4 and 5, shows an insignificant effect on the subsequent Young’s modulus which drops from 1.8 to 1.4 GPa confirming the finding made in section 5.4.3.3. Further, a specimen damaged to similar displacements (from Test No 5 to 9 with a displacement of 0.6 mm) exhibits a nearly similar stiffness ($E^*$) reaching a plateau with time. The results are a good indication of the limitations of the healing ability of the ECC material.
Figure 5-17: Tensile stress-strain relationships. Specimen successively tested and cured in water, initially tested up to different displacements (0.2 mm, 0.4 mm, 0.6 mm and 0.8 mm monthly and up to 4 months) and then to similar displacements (0.6 mm monthly and up to 11 months) until final testing being the 9th test.
5.4.3.5 Summary

The study shows that the ECC material is able to withstand further loading after being initially damaged. The magnitude of the initial damage seems to have an effect on the subsequent mechanical performance and the effect of the curing duration seems to be insignificant. However, the variability observed within the specimens made from a similar batch (as seen in Chapter 4) could not enable a direct comparison between specimens and hence a definite conclusion on the effect of the initial testing and curing duration cannot be made. The specimen being re-tested would rather form new cracks than opening existing ones, especially at lower loads, thus confirming the healing of the ECC material. More tests and data are required for statistical analysis to demonstrate the healing ability of the material. An
appropriate testing program related to the application of the ECC material in tunnel linings could be undertaken, where the material would be tested for its self-healing ability in the same conditions as when in service, this would give a better indication of its limitations for its application in civil engineering.

5.4.4 Crack formation and widths

Section 5.4.3.2 demonstrated that the initial damage may have an effect on subsequent mechanical performance although more data is needed to confirm the hypothesis, however the curing duration after initial damage seems to have an insignificant effect on further mechanical performance (section 5.4.3.3). Section 5.4.3.4 demonstrated a certain limitation of the healing ability of the material. Hence, further investigation is required to determine the possible factors influencing the self-healing ability of the ECC material such as crack widths along with possible healing mechanisms and products. ECC material in service tends to form cracks due to loading and shrinkage, hence evaluating the crack width can give a better understanding of the ability of the material to heal itself after the formation of cracks. Yang et al. (2009) stated that an important factor controlling the self-healing mechanism is the crack width which should be kept below 150 µm and preferably below 50 µm (section 2.6.5), hence suggesting that healing of the material is dependent on the crack width.

The surface of the specimen C7125-9 (as shown in Figure 5-19a) was examined at the end of testing using the Reflected Light microscope equipped with a camera (section 3.5.4). Figure 5-20 shows the surface of specimen C7125-9. The pictures reveal one crack with a value of 36.39 µm (Figure 5-20a) and the largest crack found had a width of 93 µm (Figure 5-20b). Figure 5-20 shows healed cracks remaining intact, which confirms that ECC material when being re-tested forms new cracks rather than opening existing ones as the load increases; hence indicating a certain level of healing of the material. Crack widths not exceeding a value of 100 µm are in accordance with the literature, which suggests this as a condition for self-healing of ECC materials. This is also in line with the value of crack widths linked with multiple-cracking behaviour associated with the fibre-bridging mechanism for this particular system and the subsequent pseudo-ductility for ECC material as suggested in section 2.5.2.
The measurements of crack widths are also promising if ECC material is to remain non-permeable, which is confirmed as most crack widths are below 60 µm. The largest crack observed is still below the required limit to ensure that a damaged structure does not leak. This would have a significant benefit when used as a secondary lining in hydraulic tunnels. Further analysis of the crack width along with the stress would enable a better understanding of the results observed.

5.4.5 Mechanisms of self-healing

5.4.5.1 Introduction
As presented in section 2.6.5, ECC material can heal after the appearance of cracks due to shrinkage and/or loading when the material is in service. Healing of the material can occur due to either the addition of a new component such as micro-capsules containing a healing agent or bacteria, or within the material itself, this latter process being called self-healing or autogenous healing. However, autogenous healing is likely to be the main factor considered especially in the context of a hydraulic tunnel transporting potable water, where the addition of another constituent is hardly feasible and could lead to contamination of the contents of the tunnel particularly if healing is due to microbial action. Several possible mechanisms of self-healing of ECC material are presented in section 2.6.5. These include:

- the further hydration of un-hydrated cement (especially relevant due to the high cement content present in ECC material).
- the formation of CaCO₃ or Ca(OH)₂
- the presence of impurities or loose concrete particles blocking the cracks
• the expansion of the hydrated cementitious matrix (swelling of CSH)

In this section, SEM images of cracks being healed are presented detailing the presence of unhydrated cement.

5.4.5.2 Closure of cracks

In the current study, a specimen initially tested up to the formation of initial cracks and then returned to the water bath (at 20 °C) was examined using SEM before being re-tested. Figure 5-21 shows the cracks as partly closed which demonstrates a possible healing mechanism revealed by the formation of thin bridges joining the two faces of the cracks; the bridges become thicker and thicker with time until the complete closure of the cracks occurs. Figure 5-22 presenting an EDS-SEM analysis of the composition of the material forming the bridges compared with the matrix (Figure 5-22a) reveals a higher carbon content from the material forming the bridges compared with the neighbouring zone (the matrix) with a higher calcium content instead (Figure 5-22b). Possibly the fibres bridging the cracks act as a “nucleation” site for the formation of the matrix and as a bridge for the healing of the cracking by initiating this mechanism. Further tests would be required to investigate the ECC healing mechanisms, knowing the depth and area of analysis of the EDS.

Comparing the images shown in Figure 5-21 to those presented in section 2.6.5 (Figure 2-26) in relation to the possible self-healing mechanism within the ECC material, it appears that further hydration of cement is the main self-healing mechanism.

Figure 5-20: SEM images revealing a possible crack healing mechanism (specimen C7124 made with fibre type T1 and process P2)
5.4.5.3 Unhydrated cement

As detailed in section 5.4.5.1, one of the possible mechanisms of self-healing of ECC material is the further hydration of the un-hydrated cement present within the material, which is highly probable within ECC due to the high cement content of the mixture. The backscattered electron mode of the SEM, enables the detection of the different constituents composing the ECC material, including the un-hydrated cement, based on a Z contrast, as detailed in section 3.5.3.3. Figure 5-23 shows the different elements constituting the ECC material, the fibre appearing black (low Z) while the un-hydrated cement (high Z) appears lighter.
Chapter 5 – Durability

Figure 5-22: Backscattered electron image (SEM) of a one year old specimen showing the different elements constituting the ECC material (specimen made with fibre type T2 and process P1)

Figure 5-24a illustrates the presence of un-hydrated cement dispersed in the whole area of the surface analysed. This good dispersion is seen as a great advantage for the autogenous healing of the material if submitted to wet and dry cycles. ECC material is expected to multi-crack under stress, hence when in water, the ingress of water will enable the hydration of un-hydrated cement present in the whole area of the specimen, accelerating the self-healing of the material for a better performance in the long-term. The close-up image (Figure 5-24b) exhibits the un-hydrated cement surrounded by a hydration shell, as illustrated in section 2.6.5 (Figure 2-27). Due to its high cement content compared to water, the cement grain is mainly hydrated at its surface.

Figures 5-25 and 5-26 present the backscattered electron images (SEM) at six locations of two specimens, one cured in air and another one cured in water. The un-hydrated cement content present in the specimen in air is compared to that of the specimen placed in water.
Figure 5-23: Backscattered electron image (SEM) of a one year old specimen (a) showing the presence of unhydrated cement on the whole surface analysed and (b) close-up of image of (a) showing the cement hydration shell containing the residual unhydrated cement grain (specimen made with fibre type T2 and Process P1).

The images in Figure 5-25 show a significant quantity of unhydrated cement dispersed over the whole area of the surface analysed: the unhydrated cement is evenly distributed between the sand and fibres. Obviously, the surface occupied by unhydrated cement is important, thus being available for the autogenous healing of the ECC material. This quantity is visually...
compared with that of the samples placed in water.

The images in Figure 5-26 exhibit again the un-hydrated cement being well distributed between the sand and fibres. However, its quantity is lower than observed in the specimen cured in air. This is probably due to the curing of the specimen in water promoting the ingress of water to the sample through the pores and capillaries, reducing the amount of un-hydrated cement present.

For the sample stored in water, although the quantity of un-hydrated cement is relatively low, it is still important. In addition, when in service, the material would be submitted to wet and dry cycles and therefore, the quantity of un-hydrated cement could be intermediate between that observed in Figures 5-25 and 5-26.

5.4.5.4 Summary

The study demonstrates that one of the possible self-healing mechanism of ECC material is the further hydration of un-reacted cement, especially given the large amount of un-hydrated cement present in both specimens cured in air and in water. In addition, in service, the ECC material is more likely to be submitted to wet and dry cycles promoting the hydration of un-hydrated cement. Further analysis where the quantity of un-hydrated cement is correlated to the wet and dry cycles to which the material is submitted when in service and the mechanical stresses during its service life would be valuable for the application of ECC in tunnelling.
Figure 5-24: Backscattered electron images (SEM) of a one year old specimen taken at 6 locations of 1 cm² surface of the specimen cured in air (specimen made with fibre type T2 and process P1)
Figure 5-25: Backscattered electron images (SEM) of a one year old specimen taken at 6 locations of 1 cm$^2$ surface of the specimen cured in water (specimen made with fibre type T2 and process P1)
5.5 Summary

Having previously described the properties of large sections of ECCs, the current chapter has considered durability. The long-term durability of the ECC, in particular, the ability to behave in a pseudo-ductile manner even after 150 years in service, is an important property. This is essentially dependent upon two factors i.e. changes at the fibre matrix interface, which may affect the ability of the fibres to slip and hence the ECC to multi-crack and the ability of the matrix to autogenously heal. The natural hardening of the matrix must also be taken into consideration. Whilst it has not been possible to test for significant periods of time, and whilst the value of accelerated aging must always be questioned and determined on a case by case basis, the results presented have shown that:

i. Flexure testing of naturally aged specimens shows that the pseudo-ductile behaviour of the ECC material is maintained over time.

ii. The analysis of the fibre surface chemistry shows signs of the coating being present on the fibre kept in water for nearly three years.

iii. Overall, the fibre-cement interface is relatively stable and is likely to remain for a significant amount of time.

iv. Autogenous healing is a reliable protective factor and there is some considerable evidence for this.

Whilst there are several opportunities for further work (including more in-depth chemical characterisation of the fibre matrix bond and its stability over time), a useful next step would be to submit ECCs to wet and dry cycles typical to the application of ECCs in real structures.

In the next chapter, the results associated with the shrinkage of the ECCs (the last significant area researched) are presented.
6 Shrinkage

6.1 Introduction

Shrinkage of ECCs is often associated with the formation of cracks and hence the risk of deterioration of structures and so is an important parameter to consider if this material is to find use in Civil Engineering applications. Different types of shrinkage that can occur in cement-based materials are reviewed in section 2.7. Within this chapter, drying shrinkage, referred to as “shrinkage” is evaluated as it is more severe in magnitude than the other types presented in section 2.7.3 and has been found to be responsible for the deterioration of structures (Zhang et al., 2009). Due to its relatively high cement content, ECC is expected to exhibit significant shrinkage particularly at low relative humidity. A particular concern is restrained shrinkage such as when the material is used as a tunnel lining, which can lead to significant internal stresses developing. This could promote cracking and deformation of a structure made with ECC, reducing its durability and potential longevity. As a consequence, before ECC can be used in a commercial context, it is important to understand the shrinkage behaviour of the material and the magnitude of any strains (and associated stresses) that might occur over time under different exposure conditions. By understanding the key parameters that influence the shrinkage characteristics of ECC, it may then be possible to identify steps that can be taken to reduce or otherwise control the shrinkage behaviour of thick sections of the material used in engineering application such as tunnel linings. In this chapter, the results of shrinkage measurements, carried out following the methodology outlined in section 3.7 for samples of ECC placed successively in different environments, are presented, and the influence of micro-silica on the observed shrinkage behaviour is discussed. For the commercial application of the material, it is important that the ECC has a shrinkage comparable to, or lower than normal concrete in order to limit additional design constraints. On this basis, there are two areas of interest that must be evaluated in order to find a way of controlling or reducing shrinkage:

- Environment: the curing of ECC, and particularly its curing environment at early age may have an effect on the subsequent shrinkage of the material.
- Additives: as detailed in the literature review, the addition of an additive, such as micro-silica, may help reduce shrinkage of the ECC. The experimental design method employed enabled an evaluation of the effect of micro-silica content, the order of
introduction of micro-silica and the mixing duration on subsequent shrinkage (and workability) in order to have a better understanding of ways to reduce shrinkage.

### 6.2 Effect of the environment

#### 6.2.1 Comparison: Specimens placed initially in either air or water

Following the methodology outlined in section 3.3.1, specimens of ECCs (500 x 100 x 100 mm) manufactured using Process P2 and including fibre type T1, were placed 24 hours after casting either in water or air and the axial shrinkage measured along the length of the specimen beam was measured over time. The specimens stored in water were kept at 20 °C ± 0.5 °C and those kept in air were at 20 °C ± 7 °C. The variation in the temperature of the laboratory was due to external seasonal temperature variations. Shrinkage measurements obtained at temperatures different from 20 °C were then corrected to 20 °C using a coefficient of thermal expansion for cement mortar, \( \alpha_s \) of 10 \( \mu \varepsilon/\text{°C} \) (Meyers, 1940) assuming that \( \alpha_s \) is not a function of the mortar age, as presented in section 3.7.2.

Figure 6-1 shows the shrinkage as a function of time for specimens aged in two different ways. The specimen cured in water for 47 days prior to being placed in air exhibited a shrinkage of 730 \( \mu \varepsilon \) at 57 days compared with a shrinkage of 1500 \( \mu \varepsilon \) for the specimen placed immediately in air 24 hours after casting. Zhang et al. (2009) showed that for normal concrete, the ultimate drying shrinkage strain is between 400 \( \mu \varepsilon \) to 600 \( \mu \varepsilon \) (Neville, 2000) whereas typical ECCs, under similar drying conditions (60 % relative humidity and 20 °C) exhibit shrinkage strains of 1200 \( \mu \varepsilon \) to 1800 \( \mu \varepsilon \). Aggregates are known to have a significant influence on shrinkage (section 2.7.6.2), hence shrinkage of ECCs using small aggregates compared with concrete, could be assumed similar to that of a cement paste under similar conditions. This shows that the fibres included within the ECCs do not have any effect in controlling shrinkage but given the strength and stiffness of the fibres, they may control shrinkage cracking in a similar manner to PP fibres added at much lower volume fractions to control plastic shrinkage (Branch et al., 2002). The relatively low shrinkage observed is in line with the literature confirming the important influence of humidity and temperature on drying shrinkage. It is also necessary to point out that if fully restrained, a shrinkage of 1500 \( \mu \varepsilon \) would give a stress of 20.25 MPa (if \( E_c = 13.5 \text{ GPa} \)) which implies significant cracking issues. Indeed, a strain of 730 \( \mu \varepsilon \) would give a tensile stress of 9.86 MPa, which could still be critical; this data confirms the need to understand and control shrinkage that occurs in ECC used in real world applications.
Figure 6-1: Comparison of shrinkage of a specimen cured in water prior to being placed in air with a specimen directly cured in air (effect of the environment)
It is apparent from Figure 6-1 that:

- **STAGE 1:**
  The specimen cured initially in water undergoes an initial expansion due to the incorporation of water during the hydration process. Then, on being allowed to dry, shrinkage follows the expected trend: the rate decreases with time (Yang et al., 2008). As detailed in section 2.7.3, water loss from the large capillary pores does not cause significant volume change, however volume change may happen if water is lost from the small capillary pores and subsequently from the gel pores. The results indicate that water may have been lost from the small capillary pores and from the gel pores, and part of it is irreversible. Then, when placed again in water, the specimen returns to approximately its original ‘as cast’ dimension after a short period of time but not the dimension it had at the start of the drying cycle which suggests that a slight irreversible shrinkage has occurred. As detailed in section 2.7.3, this reversible shrinkage observed means that the shrinkage was not associated with the collapse of the C-S-H gel structure, but probably mainly with the loss of water from the small capillaries pores. Neville (2000) noted that chemical shrinkage can also be responsible for the change in volume in the material, hence this could also explain why the material does not fully recover its shrinkage when re-placed in water.

As expected, the specimen exposed to air 24 hours after casting shows a significant shrinkage due to significant water loss from the “open” capillary pore system due to drying at a very early age (and hence low $\sigma$ and $E$). Cement hydration probably “stops” after only a few days and the chances of it starting again (even if saturated) are small. When placed back in water, the sample recovered a certain amount of shrinkage and seems to reach a plateau, i.e. it will expand a little bit as the C-S-H layers attempt to separate and the capillary pores fill with water (in addition, some cement hydration could occur), however a large part of the shrinkage is not recovered suggesting that the initial time spent in air had an influence on the ability of the material to recover its shrinkage resulting in some irreversible shrinkage. The experiments show that placing the material in water for a period of time after casting results in a lower shrinkage than if the same ECC is placed immediately in air 24 hours after casting.
• **STAGE 2:** The results of taking both the samples out of water again results in similar changes in dimension due to shrinkage. However, the specimen initially cured in water shows a 22% greater shrinkage than the specimen initially cured in air at 932 days (692 against 847 $\mu\varepsilon$). This is probably due to its initial curing in water giving the material more ability to loose water than the specimen initially cured in air. Once back in water, the specimen initially cured in air does not recover fully its shrinkage, indicating a greater proportion of irreversible shrinkage due to the possible irreversible precipitation and condensation of C-S-H (section 2.7.3). Also as noted in section 2.7.3, ageing is the parameter affecting most strongly the irreversible component of shrinkage and could explain the irreversible shrinkage after a long-period of time. Again as expected, the total absolute strain is still much larger in the specimen that was cured in air from 24 hours after casting, showing the advantage of an initial curing time in water.

### 6.2.2 Effect of the time spent in water on subsequent drying shrinkage

Data from Figure 6-1 was used to plot the subsequent drying shrinkage of a specimen at 87 days after it has been removed from water, as a function of the time spent previously in water, Figure 6-2. The figure demonstrates that the time a specimen spends previously in water has an influence on its subsequent drying shrinkage at 87 days, which is especially relevant as most of the drying shrinkage is estimated to occur within the first 90 days (section 2.7.3). The results show that an ECC which does not spend any time in water could exhibit a drying shrinkage in the order of $1500 \mu\varepsilon$, whereas a material cured for 50 days in water has a shrinkage of around $650 \mu\varepsilon$. However for longer times in water, this tendency seems to stabilise to $500 \mu\varepsilon$, reaching a typical value of shrinkage of a normal concrete material (Neville, 2000) despite the much lower A/C ratio of the ECC. Hence, Figure 6-2 reveals the limitations of the ECC in terms of the amount of time a specimen needs to spend in water in order to exhibit an acceptable drying shrinkage. This value is lower or equal to the one exhibited by a typical concrete as a design value, section 2.7. This data, presenting the expected subsequent drying shrinkage at 3 months depending on the time spent previously in water, is very valuable for the user and for the application of the ECC as a tunnel lining.
6.2.3 Discussion

Overall, the results from the shrinkage investigation show that an ECC cured initially in water has the ability to recover almost completely its shrinkage, even when subsequently placed in a dry environment for a long period of time. Hence, this suggests that an effective method of reducing shrinkage in thick sections of ECCs is possible by controlling the environment of the material at early ages. It is important that ECCs exhibit a limited and controllable shrinkage so that its response to wetting and drying can be reliably predicted and accounted for in the design process. For example, if placed as a secondary tunnel lining, the material could be sprayed with water in its early age, or covered with a curing membrane to prevent water evaporation during the first weeks of installation, hence reducing its shrinkage and encouraging curing of the cement pastes although this possibility is costly. For the application of ECCs in civil engineering, it is important that the material shows a shrinkage equal or lower to normal concrete material taken as a reference for design purposes. However, due to its high cement content, the ECC is likely to present a shrinkage equal to a cement paste of the order of 1500 µε (Zhang et al, 2009). Concrete presenting a lower shrinkage might be due to its high aggregate content. A typical commercial concrete presents an aggregate/cement ratio of 4-8, whereas the ECC shows an aggregate/cement ratio of 0.68, 0.67 and 0.66 for...
compositions A, B and C respectively. Neville (2000) showed in a study using a water/cement ratio of 0.4, a shrinkage of 800 µε for an aggregate/cement ratio of 3 whereas a concrete material with an aggregate/cement ratio of 7 exhibits a shrinkage of 200 µε after 6 months. Another study confirmed the possible reduction of shrinkage using mixtures with a high aggregate content (Zhang et al, 2009). In addition, as detailed in section 2.7.6.1, shrinkage is larger with a higher water/cement ratio and concrete materials tend to have a water/cement ratio from 0.4 to 0.7 (higher than ECC), hence shrinkage is expected to be slightly larger. Possibly, the presence of the aggregates may counter-balance for the effect of the water/cement ratio.

6.3 Effect of additives

6.3.1 Introduction

It was demonstrated in the previous section that shrinkage of ECCs can be reduced by controlling the environment of the material in its early ages; however this is not always possible as some construction projects require the structure to be commissioned quite quickly. This may not allow enough time to control the curing of material in its early age, for a period of time going from 1 to 2 months, necessary to reduce shrinkage. Furthermore, controlling the environment by spraying water on the material in its early ages, for example, could be costly and the feasibility depends on the specific project. Therefore, it would be commercially advantageous if an alternative way of reducing shrinkage could be found and tested. As discussed in the literature review (section 2.7.6), some specific additives have been observed to reduce shrinkage of cementitious materials; however any such additives may have a negative effect on the mechanical performance of the material. The literature review particularly identified the use of micro-silica powder, which seems to affect only slightly the mechanical performance: an increase of the compressive and flexural strength by 4 % (Maghsoudi and Arabpour Dahouei, 2007). Regarding health and safety concerns, powder micro-silica is classified as a non-hazardous material, falling into the category of nuisance dust, requiring only that an appropriate dust mask or respiratory guard to be worn; protective equipment must be selected to meet exposure and environmental conditions (Silica Fume Association, 2005).

6.3.2 Initial result incorporating micro-silica

A small percentage of micro-silica included in some ECC samples was tested (4.68 wt %;
Composition C). Above such levels, the high surface area can significantly reduce the workability (Kubens et al., 2008). Figure 6-3 presents the shrinkage measurements of specimens made according to the methodology described in section 3.7.2 and placed in air, which enables comparisons of the drying shrinkage results of specimens made using composition C with specimens made with composition B (without micro-silica powder; Table 3-1), but the fibre type and process do vary.

As might be expected, Figure 6-3 shows that the three specimens made using Composition B exhibit a similar shrinkage behaviour over time reaching a plateau value of 1500 µε. Hence, the results demonstrate that the process and fibre type do not have a significant effect on shrinkage of an ECC specimen placed in air. In contrast, comparing the shrinkage of a specimen made with composition C (containing micro-silica) with composition B, the analysis reveals a lower shrinkage value; nearly 1100 µε at 120 days compared with specimens without micro-silica, where shrinkage can go up to 1500 µε for the same age. The direct comparison with a specimen containing the same fibre type (T1) and made with a similar process (P1) shows a difference in shrinkage of about 400 µε at a similar age which suggests that the micro-silica has the ability to reduce shrinkage of an ECC.

Overall, the experiments reveal that although the shrinkage is still significant for the specimens made with Composition C, the addition of powder micro-silica contributed to its reduction. This shows its potential for usage in ECCs where the control of shrinkage is important. However, the reduction in shrinkage in Composition C are relatively small and its benefits need to be considered in terms of the cost. Further research carried out on the use of powder micro-silica such as optimising its content in ECCs and its effect on shrinkage and workability are detailed in the next section. Comparing these results with those obtained examining cure and looking at Figure 6-1, it appears that specimens placed initially in water prior to being moved to a dry environment could exhibit a shrinkage of 525 µε at 120 days, making the control of cure a better way of reducing shrinkage. Possibly combining the two effects could enhance shrinkage control and reduction.
Figure 6-3: Shrinkage comparison of specimens with and without micro-silica and placed in air after casting
6.3.3 Effects of micro-silica content, introduction of micro-silica and mixing time - Experimental design

6.3.3.1 Introduction
As detailed in section 2.7, a low shrinkage is preferred and a high workability is beneficial as it would enable an efficient placement of the mixture in its fresh state in structures. However, a high workability often suggests a high water content in the mixture, which can induce high shrinkages. Hence, evaluating the effect of different factors on these two parameters is important for the application of ECCs in civil engineering. Based on the experiments detailed previously, the effects of three factors (with two values/modalities each) on workability and shrinkage are studied. The importance of evaluating the following factors is detailed in section 3.7.3:

- Micro-silica (MS) content: 2.5 wt% and 5% wt%
- Order of introduction of micro-silica: Cementitious material + Water + Micro-silica and Cementitious material + Micro-silica + Water
- Mixing time: 5 mins and 10 mins

The effect of these three factors are evaluated on specimens cured in dry conditions (laboratory environment) at 58.2 days. The effect of each factor on workability and shrinkage is plotted on a graph and is represented by a bar. The longer the length of the bar, the more pronounced the effect of the factor.

6.3.3.2 Effect on workability
Figure 6-4 shows that MS content has an effect on workability: an increase of MS content decreases the workability of the mixture by 34.4 (± 5) mm as expected, whereas the mixing order (introduction of MS) and the mixing time have a relatively lower effect. However, introducing the MS after the water and reducing the mixing time to 5 minutes are beneficial to the workability of the mixture.

On that basis, the results in Figure 6-4 confirm that as the percentage of MS increases, the workability of the fresh mixture decreases. However, both the other factors appear to show no measurable effect on the workability when error bars are considered. The target for the
workability as stated in section 3.3.1 for the application of ECCs is 165 ± 15 mm, which is achievable with the percentage of MS considered.

![Effect of different factors on workability](image)

**Figure 6-4: Effect of micro-silica content, order of introduction of micro-silica and the mixing time on workability (BS EN 1015-3: 1999)**

### 6.3.3.3 Effect on shrinkage

Figure 6-5 reveals that the MS content would have a considerable effect on shrinkage: an increase of MS by 2.5 wt % (from 2.5 wt % to 5 wt %) reduces shrinkage by 90 (± 25.4) με, which is relatively small in comparison with 795 με, whereas the effect of the mixing order (introduction of MS in the mixture) and the mixing time would not have any measurable effect. However, introducing MS after the water addition and increasing the mixing time would promote shrinkage reduction. The error bar is ± 25.4 mm, therefore it can be considered that limiting shrinkage depends mainly on the percentage of MS and the two other factors do not significantly affect shrinkage from a statistical perspective.
6.4 Summary

Shrinkage of ECCs, associated with cracks and hence the deterioration of the material, is an important issue to be considered by designers of structures. Experiments demonstrate the possibility of reducing shrinkage of ECCs by controlling its environment at early ages. The test data provided valuable information as to what drying shrinkage to expect at about three months in relation to the time spent earlier in water. The results are promising for the application of ECCs as a lining in hydraulic tunnels.

The addition of micro-silica at a small percentage (below 5 wt. %) produced a reduction of shrinkage, as suggested by the literature. This reveals that alternatives can exist when certain tunnel projects have to be delivered immediately and where the early environment of the material cannot be controlled. An experimental design (Plackett and Burman, 1946) estimating the effect of three factors (micro-silica content, order of introduction of micro-silica and mixing time) on shrinkage and workability demonstrated that shrinkage can be reduced by increasing the amount of micro-silica, although this can reduce the workability of the mixture in its fresh state. A compromise on the workability may need to be made in order to obtain a reduced shrinkage. The decision to use micro-silica may also need to take into
account the cost-benefit of its use and availability of micro-silica. Although the cost of ECC material is considered to be in the order of 2.5 times the cost of a strain softening traditional reinforced material such as SFRC, direct cost savings are possible. ECC will be applied in relatively thin layers compared to SFRC, thanks to its higher bending capacity. Furthermore, the whole life cost can be even more reduced with the use of ECC thanks to its ability to multi-crack under stress (higher tensile strain capacity) instead of failing in a brittle manner after the appearance of a lower number of cracks, hence improving the durability of structures and reducing the need for further maintenance and/or repair as explained in section 2.2.3.

Another possible way of controlling shrinkage is the combination of the two factors: the initial curing environment of the material and the additive, both can be beneficial for the application of the material, whilst considering the cost and service time.
7 Concluding remarks

7.1 Summary
The construction sector (particularly that associated with large infrastructure projects) requires reliable materials, which may need to operate under adverse environmental conditions for a considerable amount of time. A mainstay of the sector is concrete reinforced with steel material, but there are issues when this is used for structures (such as hydraulic tunnels) which are exposed to water, as the steel tends to corrode. Alternatives are of interest and one alternative is Engineered Cement Composites (ECCs), which behave in a pseudo-ductile manner under tensile stress, thanks to the incorporation of polymeric fibres into a cementitious matrix. However, uncertainties remain regarding the mechanical performance, physical properties, long-term durability and shrinkage.

The objective of this study was to develop an ECC material with the required pseudo-ductility under stress (particularly in tension), long-term durability whilst in service and a reduced shrinkage. The physical properties such as fibre dispersion and orientation, density and porosity are also of interest: this forms the classical engineering triangle of the links between material composition and manufacturing process, microstructure and mechanical properties.

In the present study, the key focus of the work has been to take a system, that has been discussed in the literature and which has been further developed by Morgan Sindall, and produce thicker specimens than have been produced previously in order to understand their properties and usability for large structures. In this context a cementitious matrix reinforced with polymeric fibres has been manufactured using two different processes and fibre types. Specimens have been cast and tested mechanically. In addition to this the physical properties of the material have been characterised and the interface between the matrix and fibre investigated, the latter with particular attention to the durability of the material over time. The shrinkage, and its control, has also been studied.

In the following section some key findings from the current work are highlighted, and this is followed by some recommendations for further work.
7.2 Key-findings

7.2.1 Overview
In Chapter One, the overall aim of the project was summarised as understanding the link between the different parameters governing the ECC properties so as to optimise the ECC performance for the specific application of the material in tunnelling. This led to the identification of several key objectives. The key findings from the current work are summarised against these objectives:

i. demonstrate the ability to cast large sections and demonstrate the mechanical performance of these test-pieces
ii. determine the physical properties of these test-pieces
iii. determine the durability of the material, and
iv. understand the (drying) shrinkage

It was also noted that these objectives must be carried out in the light of the commercial context and the ability to use the material on site: optimisation of the material includes a consideration of the financial implications of the material and its usability. In particular, a requirement is the ability to take a material that has been prepared carefully under laboratory conditions and produce a form that can be manufactured in bulk quantities on-site under varying conditions and still give a reasonably consistent product that performs to its full potential. This work has considered the ramifications of scaling up the manufacturing process, deploying the material on site and the cost/benefit analysis of various mitigation options (e.g. the use of micro-silica to control shrinkage).

7.2.2 Composition and Processing
Process P2 (where the water is added to the mixture before the fibres) is preferred for enhancing the workability of the ECC material in its fresh state. Fibre type T1 when used with Process P2 enhances the mechanical performance (a stress up to 5 MPa associated with a strain from initial crack of more than 2.0 %). The casting method plays an important role in the mechanical performance and the fibre dispersion seems excellent with both processes.

7.2.3 Mechanical Properties
Multiple cracking was observed in both tensile samples of greater scale than tested by other researchers and in flexure, leading to a pseudo-ductile behaviour of the material under stress.
Fractographic analysis of specimens tested in tension revealed signs of detached PVA material on the pulled-out fibres, showing a certain resistance offered by the composite fibre-cement to the tensile stress and hence demonstrating the importance of the fibre-cement interface in the pseudo-ductility of ECC material. Single fibre pull-out testing from a cementitious matrix at 28 days showed the interfacial bond properties in accordance with the micromechanical model suggested in the literature as a condition for the pseudo-ductility of ECC material. The application of theoretical models to the ECC shows that the modified ACK model considering fibre orientation would be appropriate for ECC and that fibre pull-out is more likely the mechanism responsible for the pseudo-ductility of the material.

7.2.4 Physical Properties
The fibre dispersion seems excellent with both processes and investigation on fibre orientation showed an improved mechanical performance in line with a greater degree of fibre orientation. A better alignment of the fibres along the tensile axis could explain the enhanced mechanical performance of the specimens cast in a mould compared with the specimens cut from a slab. The trend of decreasing maximum strain with increasing thickness is most likely a function of increasing variability in the fibre orientation. Thin sections of up to 13 mm with fibres of length 12 mm, are more likely to lead to the manufacture of specimens with most fibres orientated in the tensile axis direction. Specimen thickness seems to control the degree of contribution of the fibres to the mechanical performance. Density and porosity measurements demonstrate that the ECC can still operate effectively despite the presence of a significant amount of porosity, and the best performance observed in tension corresponds to the specimens with the highest porosity. A high porosity tends to promote a large strain after the initial crack and a high density correlates with a higher maximum stress.

7.2.5 Durability
The pseudo-ductility of ECCs is maintained with time, for at least three years and the specimens tested did not show any evidence of its reduction. The fibre surface analysis does not show any degradation of the coating on fibres kept in water for nearly 3 years, hence demonstrating its stability. The fibre-cement interface analysis enabled the identification of the possible bond at the interface and the possible aging mechanisms. Single fibre pull-out tests at different ages revealed that the fibre-cement interface is relatively stable for up to 5 months and is likely to remain so for a significant amount of time, if not for the whole design.
life. Flexural and tensile testing of initially damaged specimens demonstrated that ECCs are still able to perform well, thanks to the ability of the material to self-heal after the appearance of initial cracks. Specimens re-tested are more likely to form new cracks rather than opening old ones. The crack widths remained below 100 µm confirming the water-tightness of the ECCs.

### 7.2.6 Watertightness and Shrinkage

Shrinkage can be reduced either by controlling the environment of the ECCs especially in its early age or by adding a small amount of micro-silica (at most 5 wt. %) whilst compromising on the workability of the fresh mixture. The control of the initial environment of the material has a bigger impact on shrinkage reduction compared with the use of additives. Combining both effects could probably be more beneficial, this could be evaluated in further work.

### 7.3 Further work

The study on the ECCs (composition and manufacturing process, mechanical performance, physical properties, durability and shrinkage) enabled the formulation of important conclusions and findings for the application of ECCs in tunnel linings. However, there are a number of areas where further work is required in order to gain a better understanding of the material. In addition to collecting more data and testing the repeatability of the behaviours observed, questions arising from the current work could be answered through further work including:

- Further mechanical tests of older specimens to demonstrate the mechanical performance over longer periods of time.
- Further investigation of the fibres and surface coating in both water and other environments (e.g. cement pastes).
- Development of a more detailed (chemical) model of the fibre-matrix interface and its evolution over time.
- Investigation of thicker sections, in particular the potential need for a higher volume fraction of fibres.
- Investigation into the application on site of this material, especially given the potential effect on workability of a higher volume fraction of fibres.
• Further work on the effect of both the environment and the use of the additive such as micro-silica on shrinkage reduction

Provided that the recommendations of this work are followed, the ECC material would be ready to be used in the real world as a tunnel lining. In the current study, a clear link is established between the composition and mixing processes, and the required properties such as the mechanical performance including the physical properties, durability and shrinkage so as to have a better control of the ECC performance for its specific application in tunnelling. Hence, in order to transition this material to other civil engineering applications, it would be necessary to define the required properties and as a consequence, adapt the parameters such as composition and processing thanks to the understanding of the influence of these parameters on the subsequent properties gained in the current study.
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APPENDIX

The paper presented at the International Conference of Composites Materials in Korea (ICCM18 - 2011) is detailed:

18th INTERNATIONAL CONFERENCE ON COMPOSITE MATERIALS

ENGINEERED CEMENT COMPOSITES PROPERTIES FOR CIVIL ENGINEERING APPLICATIONS

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Keywords: cement composite, polymeric fibres, ductility, durability, shrinkage

Abstract

Engineered Cement Composites (ECC) materials have the potential to be used in civil engineering applications where a level of ductility is required to avoid brittle failures. However uncertainties remain regarding mechanical performance, physical properties, shrinkage and durability. In the present work, specimens containing cement powder and admixtures have been manufactured following two different processes and tested mechanically. Multiple matrix cracking has been observed in both tensile and flexural tests and this leads to “strain-hardening” behaviour. The results have been correlated with sample density and porosity and it is suggested that higher levels of porosity do not necessarily lead to a loss of the strain hardening capacity. Shrinkage has been investigated and it is shown, consistent with the literature, that shrinkage can be reduced both by controlling the initial environment to which the material is exposed and by the use of additives. Durability was assessed by flexure testing of beams specimens aged for different times. Initial testing (up to one year) indicates that the specimen retain ductility, although the initial cracking threshold increases with time – which may have implications for longer aging times.

1 Introduction

Cements, which are intrinsically brittle materials, can exhibit a degree of ductility when reinforced with a sufficient volume fraction of a fibrous phase. Recent work [1] has demonstrated the potential of a particular family of these materials comprising polymer fibre reinforcement and a cementitious matrix. According to this and related studies, this ECC material (containing polymeric micro-fibres in a cement matrix) exhibits ductility under stress, instead of failing in a brittle manner. In particular, it was shown that cast, flat specimens exhibit strain-hardening, when loaded in tension, as a result of multiple-cracking of the matrix. Based on such results, it would appear that these materials have the potential to be used in civil engineering applications.

Of particular interest is the possibility of the elimination of steel from reinforced concrete ensuring that no long-term corrosion exists; this is especially relevant for structures designed to contain water.

Before this material can be used in a commercial structural context, there are a number of issues that must be addressed. These include: optimising material design and manufacturing routes (with reference to composition, fibre volume fraction and distribution, and shrinkage behaviour), demonstrating that the ductility can be achieved in different design geometries (including different length scales) and the long term durability of the structure, with particular reference to the role of the fibre-matrix interface.

The aim of the present study is to contribute to the understanding of these issues in order to facilitate the implementation of these materials. The current paper presents initial results relating to mechanical behaviour, physical properties, shrinkage and durability.

2 Materials and Manufacture

2.1 Raw materials

The constituent materials for the ECC used in the present work are cement powder, fine aggregates, water, admixtures and polymeric fibres (the latter at 2% by volume). The polymeric fibres have a nominal diameter of 40 µm and a length of 8 mm. Two types are used: Type 1 (T1) and Type 2 (T2). T2 is resin-bonded, whereas T1 is not resin-bonded.

2.2 Manufacture and Process

Small specimens were made with a Hobart commercial kitchen mixer whilst larger mixes were prepared using a concrete mixer. The different components are added successively, mixing until a homogeneous distribution is achieved before adding the next component. The order of the incorporation of a component has, in general, little effect. However, the point in the manufacturing cycle when the fibres are added has an effect on the
eventual distribution of the fibres in the cured ECC. In Process 1 (P1), fibres are added to the dry ingredients prior to the addition of water whilst in Process 2 (P2), water is added to the mix before the fibres.

3 Experimental methods

3.1 Introduction

The interesting feature of this material is its ductility, which means that structural failure by catastrophic fracture is less likely to happen. Consequently, while (cube) compression tests have been carried out on the material, the resulting values are not particularly helpful in evaluating structural performance. Therefore, flexure and tensile testing are more appropriate to demonstrate the performance of the ECC material.

In order to understand the variability in mechanical properties, it is important to appreciate the fibre dispersion and to understand the relationship between this and the density and porosity of manufactured samples. Manufactured test samples can potentially lead to preferential alignment of fibres, clustering of pores and variation in pore size. Density measurement and the other characterisation techniques used are also discussed in this section.

3.2 Tensile Testing

Thin dog-bone shaped specimens (Fig. 1) are loaded in tension using a testing machine (Instron, 5500R 4505 with a load cell of 100 kN) in a displacement control at a rate of 0.05 mm/min.

![Test specimen geometry and gripping arrangement](image)

Fig. 1. Tensile test arrangement for (a) Test specimen geometry and (b) Gripping arrangement

The flexibility to correct for imperfections in the specimen geometry and misalignment in the test machine is given by the pin situated at the top grip.

3.3 Flexure Testing

Flexural testing has been carried out based on one of the published concrete standards [2]. Beams (500 mm x 100 mm x 100 mm) were loaded in four point bending (4PB) using a testing machine (Controls, Triaxial tester T400 Digital with a load capacity of 50 kN) in displacement control at a rate of 0.2 mm/min. Fig. 2 shows the geometry and load application points [3].

![Schematic diagram of the flexure test specimen](image)

Fig. 2. Schematic diagram of the flexure test specimen

Load and strain data were used to produce Moment-Curvature plots. The curvature, $\kappa$, gives a measure of the degree of (uniform) bending in the sample and may be determined using eq. 1:

$$\kappa = \frac{\sigma - \varepsilon}{t}$$

In equation (1), the terms $\sigma$ and $\varepsilon$ denote the tensile and compressive surface strains and $t$ is the sample thickness.

Flexural testing is carried out on a range of beam specimens to evaluate the effect of process and fibre type on mechanical behaviour.

To be used in civil engineering application, the ECC material should be able to maintain its ductility with time (aging), and this is dependent on the ability of the fibres to slip in the cementitious matrix under stress. To investigate this phenomenon, flexural tests were carried out on aged samples.

The autogenous healing ability of the ECC material is also evaluated by flexural testing. Beam specimens are tested until the appearance of first cracks. They are then placed in an aqueous environment for the opportunity to heal and then retested in flexure. It will be assumed that if the
material exhibits an enhanced mechanical response on retesting (for instance a higher load at the onset of non-linearity in the load-displacement response) then the cracks have experienced some degree of healing.

3.4 Scanning Electron Microscopy (SEM)

Fractured surfaces from the tensile dog-bone samples were examined using a Scanning Electron Microscope (SEM). In this way, the matrix porosity could be visualized and preliminary observations regarding the fibre distribution and the fibre-matrix interface could be made. A Hitachi, S3200N SEM was used. Small samples were cut from the cracked faces of the tension specimens and gold coated (two 6 nm coatings of 60% gold and 40% palladium) to make them conductive for SEM examination.

3.5 Density and porosity measurements

Density and porosity measurements were made using samples cut from both tensile and flexural specimens.

The samples are weighed to determine their mass. The volume is measured using a water displacement technique for the samples from the specimens tested in flexure and by direct measurement of dimensions for the samples tested in tension.

Porosity P is determined as follows. Samples are placed in the kiln for 24 hours at 50 °C to dry and are then weighed (m_dry). The sample is then placed in vacuum to eliminate as much of the air present as possible prior to the introduction of water to occupy the volume left empty by the air. The samples are weighed again to determine m_wet. The porosity is given by:

\[ P(\%) = \left( \frac{(m_{\text{wet}} - m_{\text{dry}})}{m_{\text{dry}}} \right) \times 100 \]  

In equation (2), \( \rho_{\text{wet}} \) denotes the density of water.

3.6 Shrinkage

To be used in civil engineering applications, the ECC material may require a design life of more than 100 years, during which time it should preserve its ductility. The long-term durability of the composite material is associated with the ageing process at the fibre/cement interface and the effect that this may have on the ability of the fibre to slip in the matrix. Another important aspect of the durability/aging of the ECC is the drying shrinkage [4], and this has also been investigated here.

Shrinkage is evaluated using beam specimens (500 mm x 100 mm x 100 mm) (Fig. 3) and taking measurements along the 500 mm length. Two metallic studs are embedded in the specimen and are used to record the change in length. Shrinkage beams are manufactured by pouring mixes into a mould and demoulding after 23 hours. The beam is then placed in water for an hour. The first measurement is taken at 24 hours.

![Fig 3. Shrinkage beam specimen](image)

4 Results and discussion

4.1 Effect of process and fibre type on mechanical performance

4.1.1. Flexure test results

Fig. 4 illustrates typical moment-curvature behaviour for an ECC specimen tested in flexure: an elastic region until the appearance of the first crack followed by a region of "strain-hardening", associated with the formation of multiple cracks, leading up to the failure of the specimen.

![Fig 4. Typical bending moment – curvature response for a beam specimen tested in four-point bending](image)
The results obtained with specimens made using fibres T2 (Fig. 6) show that the specimens made with Process 1 exhibit improved mechanical behaviour compared to Process 2 (strain-deflection associated with a good maximum load), perhaps suggesting a better dispersion of fibres when they are added to the dry components rather than added to the wet mix.

**Fig. 6.** Bending moment-curvature data for specimens tested in four point bending: effect of process type (using fibres Type 2)

4.1.2 Tensile test results

The tests carried out on thin dog-bones showed more variability than the flexure tests (Fig. 7). It seems plausible that this is in part a consequence of the greater difficulties in achieving multiple cracking in tensile tests (the samples are more sensitive to misalignment and initial cracking at the shoulder of the specimen that may lead to premature failure). Aged specimens tend to exhibit a higher maximum load and a lower displacement at failure. Similarly aged specimens present different performances. On average, specimens made with fibres T2 show a similar failure load to specimens made with fibres T1 (around 6000 N in each case) but a higher displacement to failure (around 1.0 mm compared to 0.5 mm).

Hence these tests also suggest that T2 fibres give an enhanced mechanical performance – possibly the resin-bundling gives enhanced dispersion.

**Fig. 7.** Load-displacement results for specimens tested in tension: effect of fibres type and age

Figure 8 shows SEM photomicrographs of the fracture surface of sample C2105 (Fibre Type 1, Process P2). Porosity in the matrix is apparent and it is apparent that there are regions where the fibres are bundled and regions of better dispersion. These images also suggested that there was some deposit on the surface of the fibres confirming some level of interaction (mechanical or chemical) at the fibre-cement interface. From images such as these it is possible to make simple estimates of the volume fraction of fibre, which was consistent with the known levels of addition, and porosity.

**Fig. 8.** SEM images of specimen C2105

4.2 Physical properties

In this section, values of density and porosity are reported for a range of samples and compared with the mechanical properties. Table 1 shows the data for the flexural test specimens. The average density measured on eight specimens was 1862 kg m\(^{-3}\) with a standard deviation of 37 kg m\(^{-3}\). The porosity values measured on 4 samples were reasonably consistent, in the range 1.1 - 1.6%, although there is no measure of scale (large number of small pores or
small number of larger pores) or distribution (clustered, predominantly found at faces, evenly dispersed). Overall there is not sufficient variation in the parameters to draw any meaningful conclusions at this stage, other perhaps than that further testing is required.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Description</th>
<th>Maximum load (kN)</th>
<th>Curvature at ML (1 X 10^6 m^-1)</th>
<th>Density</th>
<th>Porosity</th>
</tr>
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<tbody>
<tr>
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<td>52.4</td>
<td>6.5</td>
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<td>1.7</td>
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<td>2505</td>
<td>2.5</td>
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<tr>
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<td>11.3</td>
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<td>3.3</td>
</tr>
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</tr>
<tr>
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<td>80.4</td>
<td>12.1</td>
<td>2577</td>
<td>3.9</td>
</tr>
</tbody>
</table>

Table 1: Important flexural test parameters associated with the values of density and porosity.

Table 2 shows the corresponding data for the tensile samples. The lowest porosity samples appear to have the highest failure loads, but this does not correspond to the highest displacement. The largest displacements are actually associated with the highest porosity. It is perhaps possible that higher levels of porosity may promote first cracking and subsequently multiple cracking at lower loads but that when first cracking occurs at higher loads it is more likely to lead to specimen failure with reduced associated displacement. Further experimental work is needed to test these hypotheses but it appears that the ECC material can still operate effectively (at least on a short term basis) despite the presence of significant porosity.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Description</th>
<th>Maximum load (kN)</th>
<th>Displacement at ML (mm)</th>
<th>Density</th>
<th>Porosity</th>
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<td>0.54</td>
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<td>3.3</td>
</tr>
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<td>0.64</td>
<td>2557</td>
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</tr>
<tr>
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<td>60.4</td>
<td>0.72</td>
<td>2577</td>
<td>3.9</td>
</tr>
</tbody>
</table>

Table 2: Important tensile test parameters associated with the values of density and porosity.

4.3 Shrinkage (unrestrained)

4.3.1 Effect of the environment

The results from the shrinkage investigation confirm the possibility of reducing shrinkage by controlling the environment of the material in its early age. Fig. 9 shows the shrinkage as a function of time for specimens aged in two different ways. The specimen cured in water prior to being placed in air exhibits shrinkage of 730 µε at 57 days compared with a shrinkage of 1500 µε for the specimen placed immediately in air. It is also apparent from Fig. 9 that the specimen placed immediately in water almost returns to its original dimensions.

Fig. 9. Comparison of shrinkage of a specimen cured in water prior to being placed in air with a specimen directly cured in air. These results are important for civil engineering applications, as the reduction in drying shrinkage results in a significant reduction of the risk of cracking of structures.

4.3.2 Effect of additives

Additives can also reduce shrinkage; an ECC mx which incorporated micro-silica powder resulted in a reduction of drying shrinkage by approximately 500 µε (composition C in Fig. 10).

Fig. 10. Shrinkage comparison on specimens with and without incorporation of a specific admixture.

Further work in this area will inform investigation into specific applications of this modified material.

4.4 Durability

4.4.1 Time effect on ductility

The results revealed a higher maximum load at failure when specimen is aged 307 days than when aged 28 days (Fig. 11). The curvature value at maximum load is also higher at 307 days, even though the overall ductility is reduced.
4.4.2 Self-healing

Cracks appear routinely during the life of cementitious materials under a combination of shrinkage and stress. The experiments undertaken reveal the possibility of the ECC material to exhibit ductility with time even when subject to a prior stress causing the formation of cracks in the specimen (Fig. 12). It appears possible that the increase of performance when re-loaded is due to the (partial) healing of cracks in the material.

Fig. 12. Preloading and reloading bending moment – curvature of ECC specimen

5 Concluding Remarks

This paper has provided an overview and an initial experimental investigation of a number of factors that influence the performance of ECC materials. Multiple cracking phenomena have been observed in tensile samples of a greater scale than tested by most researchers. Further work is needed to understand in more detail the roles of the manufacturing route, fibre type and fibre distribution and porosity. Control of the environment has been confirmed to be important in influencing shrinkage behaviour and there is evidence that the system can show healing after aging. With regard to understanding the aging phenomena of ECC materials in greater details, it will also be necessary to consider the surface chemistry of the fibres and the behaviour of the fibre-matrix interface with time.

Acknowledgements

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References

Engineered Cement Composite Materials Characterization for Tunneling Applications

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Abstract—Cements, which are intrinsically brittle materials, can exhibit a degree of pseudo-ductility when reinforced with a sufficient volume fraction of a fibrous phase. This class of materials, called Engineered Cement Composites (ECC), has the potential to be used in future tunneling applications where a level of pseudo-ductility is required to avoid brittle failures. However, uncertainties remain regarding mechanical performance. Previous work has focused on comparatively thin specimens; however, for future civil engineering applications, it is imperative that the behavior in tension of thicker specimens is understood. In the present work, specimens containing cement powder and admixtures have been manufactured following two different processes and tested in tension. Multiple matrix cracking has been observed during tensile testing, leading to a “strain-hardening” behavior, confirming the possible suitability of ECC material when used as thick sections (greater than 50 mm) in tunneling applications.

Keywords—Concrete composite, polymeric fibers, pseudo-ductility, test-geometry

I. INTRODUCTION

ENGINEERED Cement Composites (ECC) materials, typically consisting of a cementitious matrix reinforced with a low volume fraction of small diameter fibers, can exhibit a degree of pseudo-ductility under stress, instead of failing in a brittle manner. Previous work has demonstrated the potential of this particular family of materials [1]. According to this and related studies, specimens with a thickness of 12.7 mm have exhibited a pseudo-ductility due to a process of multiple cracking when loaded in tension [2]-[3]. Based on such results, it would appear that these materials have the potential to be used in civil engineering applications where a level of pseudo-ductility is required to avoid brittle failure. Of particular interest is the possibility of the elimination of steel reinforcing bars from concrete structures ensuring that no long-term corrosion exists, which is especially relevant for underground water-retaining structures such as hydraulic tunnels.

Deterioration arises from corrosion, especially when the steel reinforcement becomes exposed with time due to cracking. The pseudo-ductility of the ECC material associated with matrix cracking gives enhanced strain capacity before damage localization. The material, being able to exhibit pseudo-ductility under tensile stress, could be used in tunnel lining technology for repair of existing tunnels and also in new tunnel construction.

Fig. 1 illustrates a case where ECC is used as a repair material to the intrados of the original pre-cast concrete lining (PCC lining). In the case where the primary lining is considered impermeable, PCC sustains the ground load (including ground water), and in the case where the primary lining is very permeable (worst case scenario), the ECC lining will have to withstand both internal and external water pressure. The reinforcement material is also considered for the extension of the design life of existing tunnels, where there is internal water pressure.

Fig. 1 ECC tunnel lining for repair

Most of the current water tunnels operate as gravity pipelines, whereas future operational requirements will demand the water to be pumped and distributed to the other reservoirs, and therefore a high internal pressure (often up to 6 bars in the UK) will be applied. The majority of current hydraulic tunnels in the UK are inadequate to cope with the internal pressure as many of them are built as wedge-block linings and therefore a high water pressure can blow out the segments if there is not adequate ground support. The use of a permanent lining with fiber reinforced cement composite could also be an alternative for tunnels where a high internal water...
pressure is anticipated. The presence of multiple fine cracks (as opposed to a low number of wider crack openings) helps to maintain the watertightness of the water-pressurized structures in service conditions and allows self-healing to take place. The ECC mixture could be suitable for both sprayed and cast-in-place (behind a shutter) applications.

II. CONTEXT OF THE STUDY

Previous studies focused on thin specimens. For example, the Japanese Society of Civil Engineers (JSCE) reporting on High Performance Fiber Reinforced Cement Composites (ECC being a typical example of this type of material), specifies the use of a thin dog-bone shape specimen 13 mm thick (Fig. 2), with an angled shoulder between the grip region and the gauge (length of 80 mm) for testing the ECC materials in tension [4].

In practice, ECC materials might be used in structures of thicknesses of the order 50-150 mm (and potentially greater). Hence, the need to move from initial work focusing on comparatively thin sections to thicker sections is essential.

![Fig. 3 Schematic of unconfined tensile test based on the Japanese Standard [4]](image)

Thicker sections have been tested more recently, these were 20 mm [5], the thickest found in the literature for this type of material. The initial tensile testing results found a maximum strain of 4 %, lower than found in thinner section specimens showing a tensile strain above 5 % [2].

In the current work, the Japanese Standard for testing the material in tension has been followed regarding the "dog-bone" shape of the specimen, but it was considered necessary to increase the thickness specified in the Standard, to 30 mm. Additionally, it was noted that the geometry presented in the standard gives a very sharp transfer between the shoulder and the gauge. The current work has used specimens where the transfer has been made less sharp by giving the shoulder a radius of curvature of 185 mm (Fig.3).

Therefore, the specimen thickness is almost four times the fiber length (8 mm) compared with thinner sections, and when casting it is more likely that fibers will be randomly distributed in all directions: this may affect the mechanical performance of the ECC in tension.

![Fig. 2](image)

![Fig. 3](image)

Before this material can be used in a commercial context for applications with thick sections, the mechanical performance of the ECC material, especially in tension, must be demonstrated. In addition, there is capacity for optimizing material design and manufacturing routes (with reference to composition, fiber volume fraction and distribution). The link with processing and composition must also be evaluated.

The specific aim of the present study is to contribute to the understanding of these issues in order to facilitate the implementation of these materials in civil engineering applications.

III. MATERIALS AND MANUFACTURE

A. Mixture Composition

Previous work specified a mix containing cement powder, water, aggregates, admixtures and polymeric fibers at 2 % by volume [2]. In the current work, polymeric fibers have a nominal diameter of 40 μm and a length of 8 mm. Two types are used: Type 1 (T1) and Type 2 (T2). T2 is resin-bundled, whereas T1 is not resin-bundled.

B. Processing

Small specimens were made with a Hobart commercial small-scale mixer (6 liter capacity). The different components were added successively, mixing until a homogeneous distribution was achieved before adding the next component. The order of the incorporation of a component has, in general, little effect. However, the point in the manufacturing cycle when the fibers are added has an effect on their eventual distribution in the cured ECC. In Process 1 (P1), fibers were added to the dry ingredients prior to the addition of water whilst in Process 2 (P2), water was added to the mix before the fibers.
C. Casting

A study conducted on polypropylene fibers in a cementitious matrix demonstrated a clear link between the casting method, on which depend the fibers orientation and the mechanical performance [6]. It appeared that extruded specimens with approximately 80% of the fiber content aligned with respect to the extrusion direction exhibit enhanced mechanical performance compared with cast specimens having a broader distribution of fiber orientations. In the current study, the fresh mixture is poured into a mold producing dog-bone shaped specimens 30 mm in thickness. As a result, specimens of similar thickness were cast and from which dog-bone specimens were subsequently cut, using a water-jet cutting process (Fig. 4). The cutter was a Global Cutting Technologies YC-L1212 using Naiky, NeStudio™ V9 control software, cutting parameters were 150 mm/min and 100 Mesh Garnet sand.

![Fig. 4 Cutting of a test sample from a 30 mm thick plaque using a water-jet cutter](image)

IV. EXPERIMENTAL METHODS

A. Introduction

The fundamental attraction of this material is its pseudo-ductility, which means that structural failure by catastrophic fracture is less likely to happen. Tensile testing is the most severe test case for the material, whereas flexural testing is more likely to promote a graceful failure. Consequently, the tensile testing is more likely to demonstrate the performance of the ECC material and has been used for the current work.

In order to understand the variability in mechanical properties, it is also important to appreciate the fiber dispersion and to understand the relationship between this and the density and porosity of manufactured samples. Manufactured test samples can potentially lead to preferential alignment of fibers, clustering of pores and variation in pore size.

B. Tensile testing method

The dog-bone shaped specimens were loaded in tension, using a Universal Testing Machine (Instron 4505 5500R with a load cell of 10 kN capacity and using Butechelli 2 Instron’s proprietary software for control and data acquisition) in displacement control at a rate of 0.05 mm/min. The flexibility to correct for imperfections in the specimen geometry and misalignment in the test machine is given by the pin situated at the top grip. Load and cross-head displacement data were used to produce stress-strain plots.

V. RESULTS

A. Introduction

Three specimens were manufactured per fiber and process type; specimens were tested at 27 days.

First, both fiber types were tested with specimens made using Process 1. Then, as Process 2 found an advantage over Process 1 in terms of workability (the ease of placement and the resistance to segregation [7]), it was decided to test the best fiber obtained previously with both Processes to confirm the selection. An acceptable workability is very relevant when placing this material in construction. Workability is measured following BS EN 1015-3 [8]. Table I presents values of workability measured for each batch manufactured.

<table>
<thead>
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<th>Fiber/Process</th>
<th>Workability (mm)</th>
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<th>$\sigma_{max}$ (MPa)</th>
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</table>

$\sigma$ at FC = stress at first crack, $\sigma_{max}$ = maximum stress, $\epsilon$ from FC = strain from first crack, MPa = megapascal.

The effect of the casting process (specimens cast in molds and cut from a plaque) on the mechanical performance is also considered.

B. Influence of fiber type and process

Fig. 5 and Fig. 6 show an improved performance (a strain from first crack of almost 2.5%), associated with multiple fine cracks within the gauge length. Variations within similar specimens remain; this is not unexpected and is typical behavior of a ceramic. It seems also plausible that this is in part a consequence of the variability in the microstructure linked with the manufacturing process.

Comparing results (Fig. 5), it would appear that when Process 1 was used, specimens manufactured using fiber T2 exhibit a stiffer response associated with a higher maximum
stress ($\sigma_{\text{max}}$) whereas fiber T1 specimens present a higher ultimate strain. Possibly, fiber T2 promotes enhanced matrix strength due to the resin-bonding, whereas fiber T1 specimens exhibiting first cracking at low stresses enable a higher strain after first crack. These initial results demonstrate that fiber T1 would have an advantage over fiber T2 when using Process 1.

Looking at Fig. 6, specimens containing fiber T1 and manufactured with Process 2 seem to present the highest ultimate strain associated with an even higher maximum stress (up to 5 MPa) compared with specimens manufactured with Process 1. It seems that fiber T1, as in Fig. 5, promotes a higher strain and the use of Process 2 enables a higher stress. Process 2 enables, perhaps, a better fiber dispersion and therefore better mechanical performance.

![Fig. 5 Stress-strain results for specimens tested in tension: effect of fiber type (Process 1)]

![Fig. 6 Stress-strain results for specimens tested in tension: effect of process type (Fiber type 1)]

However, it should also be noted that there is still variability in mechanical performance within batches (similar fiber type and process) and understanding these variations is also part of the study.

C. Influence of casting in a mold or in a plaque

It is possible that the casting method would have an influence in physical properties and fiber dispersion and orientation, where the mold, due to its smaller size compared with the plaque, would align the fibers following the tensile axis and promote better tensile performance. The difference in mechanical performance between samples cast in molds and the ones cast in plaques are currently under investigation. The idea is to also reproduce high performance specimens, even when the specimens are cut from a plaque where they are cast.

To have a better understanding of this, a study of the physical properties, fiber dispersion/orientation will also be undertaken.

VI. CONCLUDING REMARKS

A. Summary

The pseudo-ductility of ECC's under stress is associated with the formation of multiple fine cracks in the specimen, instead of a single crack leading to brittle failure.

Based on the literature review, a specific composition, containing polymeric fibers of a diameter of 40 µm, has been selected for the study. Initial methodologies are established for characterizing the ECC material as well as for testing in tension, based on a Japanese Standard.

Mechanical testing of specimens with a thickness of 30 mm reveals promising results in tension as the pseudo-ductility associated with multiple cracks under stress is demonstrated in most of the specimens tested. These results have enabled the evaluation of specific parameters such as fiber type and process, as enhancing the mechanical performance. The casting method would play a role in the mechanical performance. However, more data, along with statistical analysis are required to support these conclusions.

Further work will concentrate on acquiring more data from specimens tested in tension, so as to validate the hypotheses regarding the difference in mechanical performances obtained when using a specific fiber type and process, the influence of the casting method and ways to optimize the mechanical performance.

B. Key-findings for tunneling applications

The current paper has provided an overview and the important parameters to consider before using ECC in future tunneling applications. The study revealed:

1. ECC material could be used in large thicknesses in structures and still perform well under tensile stresses: a pseudo-ductile behavior associated with the formation of multiple cracks, sustaining a tensile stress of up to 5 MPa (typical of a high performance concrete)

2. Process 2, where the fibers (T1) are added to the mixture after the water addition and at the end, is preferred as offering a better workability of the ECC material in its fresh state, and therefore a better ease of placement.

3. The casting method or the method of placing the ECC material in structures should be carefully chosen, as it could play an important role in the mechanical performance of the material by controlling the fiber dispersion and most probably the fiber orientation.

4. Variability in mechanical performance remains and reducing this variability should be a future priority.
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Tensile Characterisation of Thick Sections of Engineered Cement Composite (ECC) Materials

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Abstract
Engineered Cement Composite (ECC) materials have the potential to be used in applications where a level of pseudo-ductility under tensile stress is required. Most previous work has focussed on comparatively thin specimens. For future civil engineering applications however, it is imperative that the behaviour of thicker specimens is understood. In the present work, specimens containing cement powder, water, polymeric fibres and admixtures were manufactured following two different processes and tested in tension. Multiple matrix cracking was observed during tensile testing, leading to a pseudo-ductile behaviour. Complementary measurements of sample density and porosity suggest that a high porosity could be linked with an enhanced tensile strain to failure whereas high density is associated with a high maximum stress. The fibre dispersion, assessed by Scanning Electron Microscopy, indicated that mechanical performance was enhanced with increasing proportion of fibres aligned along the tensile test axis and this orientation can be linked to the manufacturing process.

Key-words: cement composite, polymeric fibre, dog-bone specimen, mechanical performance, pseudo-ductility, fibre orientation

1. Introduction
Cementitious materials are intrinsically brittle materials but can exhibit a degree of pseudo-ductility when reinforced with a sufficient volume fraction of a fibrous phase. Recent work has demonstrated the potential of a particular family of such materials, called Engineered Cement Composites (ECCs), consisting of a polymer fibre reinforcement and a cementitious matrix [1]. Table 1 presents a comparison of the performance of ECCs with other fibre reinforced cement composites [2]. According to this information and related studies of cast, flat specimens (with a thickness of 12.7 mm), ECCs can routinely exhibit significant pseudo-ductility under tensile stress, as a result of multiple-cracking of the matrix, rather than failing in a brittle manner [3,4]. In Fig. 1, it can be seen that, instead of the catastrophic failure at extremely low strains demonstrated by a traditional cement mortar, much larger strains to failure are observed. This behaviour is due to the ability of the material to crack in multiple locations such that the strain is distributed amongst a large number of cracks which typically remain sub-100 μm in width prior to final failure [3].

Based on such results, it would appear that fibre reinforced cementitious materials have the potential to be used in civil engineering applications, where the material is required to withstand a certain level of tensile stress without developing a single (or limited number of) wide crack(s). For example, the use of steel fibre reinforced concrete is now well established in the construction
community [5] and can significantly increase the ability to control crack widths at large strains. However, like traditional steel reinforcement, such fibres are prone to corrosion and because of the small diameter of the fibre, even mild corrosion can represent significant loss of cross-section and hence it becomes necessary to use more easily stainless steel fibres. There is therefore, within industry, an interest in alternative solutions that incorporate non-metallic (polymer) fibre systems [6]. ECC materials represent a natural extension of that approach but before they can be used in a structural context, there are a number of issues that must be addressed. These include optimising material design and manufacturing routes (with reference to composition, shrinkage behaviour, fibre volume fraction and fibre distribution); demonstrating that the pseudo-ductility can be achieved in different design geometries (including different length scales) and the long-term durability of the structure, with particular reference to the stability of the fibre-matrix interface. Some of these issues have been addressed [7, 8].

Perhaps one of the most useful properties of ECCs is their ability to undergo multiple cracking, a process in which the polymer fibres (at about 2% by volume) are able to bridge the crack and carry the load without failure. The cracks open minimally and instead of failure, the component continues to carry the load with the formation of multiple cracks. Counter-intuitively, it is necessary to introduce defects (to act as stress concentrators) in order to achieve the maximum crack density possible and hence increase the strain capacity. The normal defect population would of course consist of porosity whilst an engineered defect population could be introduced in addition to this [9]. However, this has potential consequences for the permeability of structures manufactured from ECCs, even if the structure is un-cracked. This is because the engineered defect population may act to transport fluids through the section, particularly when such defects represent quasi-connected porosity. In a water carrying structure, such behaviour could represent a significant limitation. In the current context, the focus is on applying ECCs in an industrial application where the achievable strain is a function of the crack density and the crack width.

Previous studies on the properties of ECCs have tended to focus on thin specimens. For example, the Japanese Society of Civil Engineers (JSCE) reporting on High Performance Fibre Reinforced Cement Composites (ECCs being a typical example of this type of material), specifies the use of a thin dog-bone shaped specimen for testing ECC materials in tension (Fig. 2). The specimen is 13 mm thick, with an angled shoulder between the ends (with a width of 60 mm) and the 80 mm gauge length having a width of 30 mm [10]. In practice, ECC materials might be used in structures of thicknesses of the order 50-150 mm. Hence, there is a need to move from initial work focussing on comparatively thin sections to thicker sections. Whilst thicker sections have been tested [11, 12], further work is still required to understand thickness effects and the effect of fibre orientation on strength.

Following this introduction, the paper is structured as follows. Initially, the materials and manufacturing methods are discussed, with emphasis on the process methodology (order of addition of constituents) and casting method, as these both have an effect on fibre dispersion and orientation. The tensile testing methodology is then presented with initial results, a subsequent refinement to the test geometry, and further testing. Mechanical property data are supported by fractography. The physical properties of the samples (density and porosity) are presented, followed by image analysis of the fracture surfaces to assess fibre dispersion and to quantify fibre orientation. Finally, the
results are summarised and the key-findings and implications of this work for civil engineering applications are outlined.

2. Materials and Manufacture

Previous work specified a mix containing cement powder, water, aggregates, admixtures (acting as rheology modifiers) and polymeric fibres at 2 % by volume [3]. In the current work, a similar composition has been employed, but here it has been optimised for industrial applications (Table 2). Two types of fibre were used (Table 3): each has a nominal diameter of 40 μm and a length of 8 mm. Both fibre types are supplied by Kuraray, the difference being that Type 1 REC15/8 (T1) is ‘oiled’ (a surface treatment to control the interface chemistry) and Type 2 RECS15/8 (T2), is the same fibre but resin-bundled. Data from the supplier (Kuraray) indicate that the Young’s modulus and tensile strength of the fibre are 40 GPa and 1560 MPa respectively.

The material was processed using a Hobart commercial small-scale mixer (6 litre capacity) following two different processes (Table 3). The different components were added successively, mixing until a homogeneous distribution was achieved before adding the next component. The point in the manufacturing cycle when the fibres are added was found to affect the distribution in the cured ECC. In Process 1 (P1), fibres were added to the dry ingredients prior to the addition of water whilst in Process 2 (P2), water was added to the mix before the fibres. It should be noted that Kuraray recommends only Process 1 in conjunction with T2 fibres. This was borne out in the initial testing which showed that the dispersion of the resin bundled fibres was very poor with Process 2. Under these circumstances, this combination was not considered further.

In order to deploy ECCs in real engineering situations (e.g. tunnel lining applications), there is a need to understand the workability of the mix, i.e. the associated ease of placement and its resistance to segregation [13]. Hence the workability of the mixes produced in this work was measured using a flow table following BS EN 1015-3 [14]. In this method, the fresh mixture is spread on a disc and the extent of spread is used to indicate the workability. Table 4 presents values of workability measured for each batch manufactured.

For the purposes of mechanical characterisation, the Japanese Standard [10] for testing ECCs in tension has been followed but it was considered necessary to increase the thickness of the “dog-bone” specimen specified in the Standard, to 30 mm (Fig. 3). Therefore, the specimen thickness is almost four times the fibre length, which is 8 mm, compared with thinner sections where the fibre length is closer to the same scale as the thickness. Hence, when casting thicker specimens it is more likely that the fibres will be randomly distributed in all directions and this may affect the mechanical performance of the ECC in tension.

A previous study on polypropylene fibres in a cementitious matrix demonstrated a clear link between the casting method (which affects the fibre orientation) and the mechanical performance [15]. It was observed that extruded specimens with approximately 80 % of the fibre content aligned with respect to the extrusion direction exhibit enhanced mechanical performance compared with cast specimens having a broader distribution of fibre orientations.
The fresh mixture is poured into a mould producing dog-bone shaped specimens 30 mm in thickness. As a comparison, thin slabs of similar thickness to the dog-bones were also cast and from which dog-bone specimens were subsequently cut, using a water-jet cutting process. The cutter was a Global Cutting Technologies TC-L1212 using Naiky, NeStudio™-V9 control software; the cutting parameters were 150 mm/min and 100 Mesh Garnet sand.

3. Tensile testing

Tensile testing is the most severe mode of loading for the material, whereas flexure is more likely to promote a graceful failure. Consequently, the performance of the ECC material in tension is critical and has been used in the current work, notwithstanding the inherent challenges in testing a material with such a brittle matrix in tension. Initially, tensile testing was carried out using a specimen geometry (see Fig. 3) based on the dog-bone of the Japanese standard.

Specimens were loaded in tension, using a Universal Testing Machine (Instron 4505 5500R, with a load cell of 10 kN capacity and using Bluehill 2, Instron’s proprietary software for control and data acquisition) under displacement control at a rate of 0.05 mm/min. The flexibility to accommodate imperfections in the specimen geometry and misalignment in the test machine is provided by the pin situated at the top grip. Load and cross-head displacement data were used to produce stress-strain plots. Using this test method, the strain to first cracking is an overestimate due to the effect of load-train compliance, but because the subsequent multiple cracking occurs at a near constant stress, the measurement of strain capacity after the first crack is accurate.

Initial results indicated that there was limited pseudo-ductility after the initial crack (Fig. 4a) when compared with the results reported in the literature [3]. Moreover, the specimens always failed at the sharp edges (as shown in Fig. 4b). This could prevent extensive further multiple cracking within the length of the specimen, and hence true pseudo-ductile behaviour is not observed.

An analysis of the tensile stress distribution within the thin dog-bone specimens under a tensile load was conducted using finite element analysis FEA (Abaqus 6.10-1). As expected, FEA confirmed the existence of stress concentrations at the transition from the sharp corner at the shoulder to the gauge length on the original geometry. The analysis suggested further that dog-bone shaped tensile specimens with a curved shoulder exhibited a reduced concentration of stress. On the basis of further analyses, a different geometry was used with a uniform radius of curvature of 185 mm, reducing the concentration of stress (Fig. 5).

Having modified the test geometry, further tensile testing was carried out to evaluate the effect of process and fibre type on mechanical behaviour. Table 3 shows the experimental test matrix relating to the fibre and process. Three specimens were manufactured per fibre and process type; specimens were tested at 27 days. The results associated with the new geometry (Fig. 6 and Fig. 7) show an improved performance in terms of strain capacity (failure strains of up to around 2.5 %), associated with multiple fine cracks within the gauge length. Variations between similar specimens remain; this is not unexpected and is typical behaviour of (cured) cementitious material. It is highly likely that this is in part a consequence of the nature of the cementitious matrix made partly of crystals and the variability in the microstructure linked with the manufacturing process as well as the presence of porosity. Fig 8 illustrates the variability in the capacity of dog-bone samples of ECC tested in
tension to undergo controlled multiple cracking. The arrows indicate clusters of cracks. As expected, the specimen that shows most cracking prior to failure also shows the greatest strain-to-failure (here taken from the point at which the first crack occurs), and the least cracking corresponds with the smallest strain-to-failure. Comparison of the samples with load-displacement (stress-strain) responses, see Fig. 6, indicate that the formation of these clusters correlate with one or more load-drop events.

Comparing the results in detail, Fig. 6 shows that when Process 1 was used, specimens manufactured using fibre T2 exhibit a higher maximum stress ($\sigma_{\text{max}}$) whereas specimens using fibre T1 show a higher ultimate strain. This is likely to be a result of a different fibre-matrix interface in the two systems and/or a different fibre dispersion and distribution caused by the rigidity of the resin-bundling, which is consistent with the literature. Possibly fibre T2 promotes enhanced matrix strength due to the resin-bundling, whereas for fibre T1 specimens, first cracking at low stresses enables a higher strain after first crack. These results demonstrate that fibre T1 would have an advantage over fibre T2 when using Process 1 promoting a larger strain to failure.

Looking at Fig. 7, specimens containing fibre T1 and manufactured with Process 2 present the highest maximum stresses (up to 5 MPa) and comparable ultimate strains to those manufactured with Process 1. It seems that fibre T1, as in Fig. 6, promotes a higher strain and the use of Process 2 enables a higher maximum stress. Process 2 enables, perhaps, a better fibre dispersion/orientation and therefore better mechanical performance. Based on the results from these experiments, performance is optimised using fibre T1 with Process 2.

Fig. 9 compares the mechanical performance of specimens cast using the modified dog-bone mould and specimens of the same geometry excised from a slab. The cast specimen exhibit both superior strength and strain to failure compared with those excised. A relatively low maximum stress (up to 3 MPa) and strain after the first crack (up to 0.2 %) are presented by the specimens cut from the slab compared with the ones made in a mould (maximum stress up to 4.3 MPa and strain after first crack up to 2.25 %). This suggests that the casting method could influence the physical properties, as well as the fibre dispersion and orientation. The use of the mould, due to its smaller size compared with the slab, could align the fibres in the loading direction and hence promote tensile performance. In contrast, the specimen cut from the slab would lead to a more random distribution of fibres and where fibres present at the edges are cut, these fibres would be expected to contribute less to the mechanical performance. This is discussed further in section 6.2.

Fig. 10 compares the mechanical performance as obtained in this study (using a specimen thickness of 30 mm) with data obtained in previous studies using different thickness section specimens and using fibres of a similar length and composition [3, 11, 12]. It appears that ultimate strain decreases to reach a plateau whereas the maximum stress slightly increases or remains constant with increasing specimen thickness.

4. Fractography
Fractured surfaces from the tensile samples were examined using a Scanning Electron Microscope (SEM); the instrument used was a Hitachi S3200N. Small samples (10 mm x 10 mm x 20 mm) were cut from the cracked faces of the tensile specimens and gold coated (four coatings of 99.9 % gold
each 2 mm thick) to make them conductive in the SEM.

The mechanical performance of the ECC material is often characterised by the ability of the fibre to pull-out within the cementitious matrix under tensile load instead of breaking. According to the literature, the fibres used in this study (REC15/8) have been specifically tailored to pull-out rather than break when ECC samples are loaded in tension [1, 16-17]. The fibre characteristics demonstrate the link between the frictional bond, the fibre surface coating content and the fibre tensile stress capacity. Another study examined fibres at different stages of pull-out from a cementitious matrix [18]; this is relevant to mechanical performance of ECC in tension. At large strains, close to failure, the ends of the pulled-out fibres exhibit thinned "valleys" of missing PVA on its surface and the embedded end is narrower than the original [18].

In the current study, the fracture surface of specimen C6543 (see Fig. 7) was examined using the SEM. An overall perspective is given in Fig. 11a which shows a cluster of fibres from the fracture surface, whilst Figs. 11b-d present examples of particular behaviour. Fig. 11b corresponds to a full fibre with some PVA material detached from its surface; this type of fibre could contribute well to the pseudo-ductility of the ECC composite. Fig. 11c provides evidence suggesting that the fibre-matrix bond-strength is, in places, greater than the strength of the cementitious matrix, leading to a cohesive failure of the matrix. This type of behaviour may contribute in a limited way to the pseudo-ductility of the composite [18]. Finally, Fig. 11d also shows that PVA material is missing, but here the fibre end appears narrower compared with its original shape; this type of behaviour is usually seen at the end of testing and will make only a small contribution to the pseudo-ductility of the ECC in tension [18].

5. Physical properties

The tensile testing results show that the thicker material is capable of pseudo-ductility, although variability in the mechanical performance remains, even between specimens from a similar process and fibre type. In order to understand the variability in mechanical properties, it is important to appreciate and understand the relation between these and certain physical properties of the manufactured samples. Density and porosity measurements were made using samples cut from the tensile specimens, enabling a direct comparison of the mechanical performance with the physical properties.

The density of the samples was measured according to BS 12390-7: 2009 [19]. The water saturated surface-dry samples were weighed in air to determine their mass (Wv). The volume was measured using a water displacement technique.

There is no British Standard for evaluating the porosity of concrete specimens. A study comparing the ASTM saturation techniques for measuring the permeable porosity of concrete, recommended the use of the vacuum saturation technique as the most efficient method [20]. Porosity was therefore determined as follows. Samples were placed in the kiln for 24 hours at 50 °C to dry and were then weighed to determine the oven dry mass in air (Wd). The sample was then placed under vacuum to eliminate as much of the air present as possible prior to the introduction of water to occupy the volume left empty by the air. The samples were weighed again to determine the buoyant mass of the specimen in water (Wb). The permeable porosity (PP) is given by [20]:

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Values of density and porosity are shown for a range of samples and compared with the mechanical properties in Table 4. The average density of twelve specimens was 1947 kg.m$^{-3}$ with a standard deviation of 23 kg.m$^{-3}$. The measured values of open porosity were on average 6.2% with a standard deviation of 1.7%, although there is not any measure of scale (large number of small pores or small number of larger pores) or distribution (clustered, evenly dispersed, predominantly found at faces).

Table 4 shows that the specimens from the mould with the largest ultimate strains ($\varepsilon$), specimens C6523 and C6543 also have the highest porosities, 7.8% and 9.5%, which is consistent with the literature [9]. The highest density specimen with a high porosity (specimen C6523) shows a large strain after first crack and a high maximum stress. The highest porosity specimen (specimen C6543) associated with an average density presents the highest strain after first crack, but a lower maximum stress. The lowest density specimens (cast in slabs) are generally associated with a low maximum peak stress ($\sigma_{\text{max}}$) and also a low plastic strain after first crack, so giving a very low pseudoductility.

Although there is variation, the data are consistent with the idea that a high porosity promotes a large strain after the initial crack and also a first crack at a lower stress whereas a high density, leads to a high maximum stress. A first crack at lower stress in high porosity specimens could be explained by the possibility that porosity may be connected with the initial flaw [9]. It is perhaps possible that higher levels of porosity promote first cracking and multiple cracking at lower loads. Whilst the trend is not conclusive, it appears that the ECC material can still operate (at least on a short term basis) despite the presence of significant porosity. The variability in the physical properties (especially porosity) and mechanical performance of specimens made from the same batch suggests that a way has to be found to control the properties of the material cast on site in order to make the mechanical performance more consistent if this material is to be used in challenging commercial situations.

6. Fibre orientation
6.1 Measurements of fibre orientation

The fibre dispersion within the cementitious matrix will play an important role in the mechanical performance of ECC materials [21]. However it is also important to consider the fibre orientation; this orientation can affect the measured properties and hence the manufacturing routes that lead to preferential alignment of fibres need to be understood. Alignment would be beneficial if the fibres need to withstand a single or dominant principal stress. However, where a multi-axial stress state exists, fibres oriented in more than one direction will be needed.

The fibre dispersion and orientation was analysed by examining samples (50 mm x 30 mm in area) cut from near the fracture surface of the specimens tested in tension. The samples were first prepared for examination and then analysed using the SEM. The examination of the fibre dispersion and orientation requires a specific sample preparation for the fibres to be identified easily. Further, the identification and quantification of various phases of the cementitious matrix require the sample
to be prepared well and usually a flat surface is preferred [22]. Several stages are required to obtain a perfectly flat and smooth surface for examination. In the present work, flat and smooth surfaces were prepared as follows:

- The cut sample was placed in a mould (such that the cut face could be polished), then vacuum-filled with an epoxy resin, which also filled the pores of the material. The resin stabilises and maintains the microstructure, enabling the sample to withstand grinding and polishing without alteration. It prevents particles plucking, pitting and micro-cracking the polished surface.
- The epoxy having cured overnight, a series of grinding and polishing steps with progressively finer abrasives (Table 5) were used to produce a flat polished specimen. A high quality finish is necessary to facilitate the identification and quantification of various phases of the cementitious material using the backscattered electron mode of the SEM [22].
- The polished specimens were then sputter coated with a film of conductive material (as detailed in section 4) to prevent the presence of electric charges when the specimen is scanned by an electron beam.

The samples cut from thin dog-bone specimens tested in tension and prepared as outlined above were examined using the SEM. The fibre dispersion and orientation were analysed in backscattered electron mode; the different elements composing the images were visualised based on a Z-contrast (the lower the atomic mass the darker the image appears, whilst conversely the higher the atomic mass, the lighter the image). The fibres are recognised by their dark colour (relatively low Z as mainly composed of carbon), their size and shape.

Fibres can be classified and categorised, depending on their orientation [21]. In the current work, approximately 300 images were taken for each of three specimens: C6523, C6543 and C6544C. The fibres are then counted and classified depending on their orientation using ImageJ (open source digital image analysis software, ImageJ 1.46/Java 1.6.0_20 (64-bit)). Fig. 12 shows a typical backscattered electron image; a circular fibre shape is perfectly aligned with the tensile testing axis whereas an elliptical shape suggests a certain orientation of the fibre - the greater the elongation of the ellipse, the larger the angle of inclination of fibre to the specimen loading direction.

Fig. 13a compares the quantity of fibres for each orientation for tensile specimens cast in a mould (C6523 and C6543) and a specimen cut from a slab cast in a similar way (C6544C). These initial results suggest a greater proportion of fibres aligned with the tensile axis (0 - 30°) for a specimen cast in a mould, especially C6523, whereas the specimen cast in a slab (C6544C) seems to exhibit a more random orientation of the fibres. As might be expected, and as can be seen in Fig. 13b, the sample with the greatest maximum stress and strain from first crack is the one with the highest proportion of fibres aligned in the direction of the applied stress. Indeed, specimen C6523, exhibiting enhanced performance, shows more than 50% of its fibres aligned along the tensile testing axis (0 - 30°) compared with specimen C6544C presenting a random orientation of fibres and lower mechanical performance. This suggests that Process 2, where the fibres are put last in the mixture, may lead to fibre alignment along the tensile testing axis when casting in a mould.

As demonstrated in the current study, fibre orientation plays a major role in the mechanical performance. Therefore, a higher maximum stress is expected with thinner specimens where the
fibres would more likely align in the tensile loading direction. Instead, the specimen thickness seems to have only an impact on the maximum tensile strain and further pseudo-ductility which seems to decrease with the specimen thickness.

6.2 Simple models for mechanical performance

Thick ECCs present considerable challenges in terms of developing predictive models for mechanical performance. Given the unresolved issues around modelling the development of damage and failure in continuous fibre laminated composites [23], it is clear that ECCs present further levels of complexity in terms of the nature of the reinforcement – a low volume fraction of partially aligned short fibre - and the uncertain role of matrix defects. Aspects of the multiple cracking behaviour may be amenable to modified versions of the classic ACK type of approach, as has been explored to some extent in papers by Li and co-workers [24] for less thick (and with correspondingly better fibre alignment) ECCs than those tested in the present work, but this requires further investigation. Instead, as a starting point, in the present section we assess simple strength models based first on fibre fracture and secondly on fibre pull-out.

Based on classical short fibre composite theory, the composite stress is related to the fibre stress (σf), the matrix stress (σm) and the corresponding volume fractions (Vf and Vm) according to:

\[ \sigma_c = \eta_f \eta_m V_f \sigma_f + V_m \sigma_m \]  

(2)

The fibre length distribution factor (\( \eta_f \)) can be calculated using the Cox equation [25], which is based on the assumption of elastic load transfer between the fibre and the matrix:

\[ \eta_f = 1 - \left[ \tanh(\beta L/2) \right] / (\beta L/2) \]  

(3)

where

\[ \beta = \frac{[2\pi G_m]}{[E_f A_f \ln(R/R_f)]} \]  

(4)

where L, Gm, Af, Rf, R are the fibre length, the shear modulus of the matrix, the cross-sectional area of the fibre, the radius of the fibre and the mean separation of the fibre.

The fibre orientation distribution factor (\( \eta_\theta \)) can be calculated using the Krenchel equation [26]:

\[ \eta_\theta = \sum_{i=0}^{i=100} \frac{V_{f_i} \ cos^4 \theta_i}{\sum_{i=0}^{i=100} V_{f_i} \ cos^4 \theta_i} \]  

(5)

In order for the cement composite to show multiple cracking and hence pseudo-ductility, the fibres have to be able to support the load on the composite, after initial matrix failure. Provided that this happens, the composite strength can be estimated from the fibre strength (\( \sigma_{fu} \)) according to:

\[ \sigma_{c,max} = \eta_f \eta_\theta V_f \sigma_{fu} \]  

(6)
Data in Fig. 13a are used to calculate $\eta_0$ for each specimen and the values are shown in Table 6. The greater fibre orientation in specimen C6523, which is apparent in Fig. 13a, leads to values for $\eta_0$ of 0.51; the value for C6543 (0.37) is close to the theoretical result for a random planar orientation (0.38) while the result for C6544C (0.31) suggests that there is a degree of orientation, but that it is not aligned with the direction of testing. The improved mechanical performance of C6523 is in line with this greater degree of orientation (Fig. 13b).

Of these three specimens (30 mm thick), C6523 was cast in a mould using Process P2 which has the greater workability. Consequently the alignment of the fibres in this sample with the mould direction seems reasonable. Specimen C6543 was also cast in the mould but using the lower workability process P1 and hence there is less alignment. With specimen C6544C which was cast in the slab using process P1, it is possible that the casting method introduced a degree of alignment and the low workability of the mix leads to this alignment remaining in the specimen cut subsequently from the slab presenting a slight fibre alignment in the opposite direction to the tensile load.

It will be recalled that the nominal volume fraction for all the materials in the present work is 2%. Hence, strengths for each system can be calculated using equation (6). The results are shown in Table 6 and vary from 9.6 MPa up to 15.8 MPa for more aligned system. These values are higher than the strengths achieved in practice, which do not exceed 5 MPa: moreover the experimental strength of the more aligned system is not significantly higher. These calculations confirm the capacity of the fibres to bridge the matrix cracks, but the over-estimate of strength suggests that a model based on fibre pull-out from a frictionally bonded interface is likely to be more appropriate.

For a nominal fibre length of 8 mm, the mean fibre pull-out length $\bar{L}_{po}$ is 2 mm, independent of the fibre orientation. Assuming that the fibre matrix interface can be characterised by a constant interfacial shear strength $\tau$, then the load to pull-out a single aligned fibre, $P$, is simply given by:

$$P = \pi d \bar{L}_{po} \tau$$

Hence, the corresponding stress on the composite to initiate pull-out failure can be estimated from:

$$\sigma_{po} = V_f \frac{P}{\pi r^2} = \frac{4V_f \pi \tau \bar{L}_{po}}{d}$$

The fibre diameter is 40 μm and the frictional shear strength is taken as 1.5 MPa, which is consistent with the literature [1] and measurements made as part of the present study using a single fibre pull-out specimen. Substitution into equation (8) gives a strength of 6 MPa. Assuming that the fibre orientation modifies the strength by pull-out in a similar way to the strength when controlled by fibre failure, then the predicted strengths for systems that fail by pull-out are in the range 1.9 – 3 MPa. In fact, using the same orientation factors for pull-out and fibre controlled failures is unlikely to be appropriate. Fibres with greater misalignment are likely to contribute more to pull-out strength than the fibre-based strength and hence the range 1.9 – 3 MPa is likely to be a lower bound, while 6 MPa will represent an upper bound. Overall the results from the pull-out model are consistent with the experimental data. Agreement is satisfactory, notwithstanding the simplifications made, such as ignoring the random nature of the fibre distribution and the role of defects such as porosity.
Noting also that based on considerations of fibre pull-out, the critical opening displacement of the crack that controls fracture should be of the order of the mean pull-out length. Based on a specimen gauge length of 200 mm, this gives a nominal strain of 1%, which is around half of the total strain observed in a typical specimen after first crack. The balance of the strain is presumably associated with the opening of the other cracks.

7. Concluding remarks

The construction sector particularly that associated with large infrastructure projects requires reliable materials: these may need to operate under adverse environmental conditions for a considerable amount of time. A mainstay of the sector is reinforced concrete with steel material, but there are issues when used for structures which are exposed to water, as steel tends to corrode. Alternatives are of interest and one such alternative is Engineered Cement Composites ECCs, which behave in a pseudo-ductile manner under tensile stress, thanks to the incorporation of polymeric fibres in a cementitious matrix. Such behaviour is associated with the multiple cracking of the matrix.

The current study has led to a number of findings:

1. Specimens with a thickness of 30 mm are able to demonstrate pseudo-ductility when tested in tension, where the pseudo-ductility of the material is associated with the formation of multiple cracks (with typical crack widths less than 100 μm). It seems on average that a high porosity promotes a large strain after the initial crack whereas a high density correlates with a higher maximum stress. ECC material could therefore be used in large thicknesses in structures and still perform well, sustaining a tensile stress of up to 5 MPa (typical of a high performance concrete). The trend of decreasing maximum strain with increasing thickness is most likely a function of increasing variability in the fibre orientation. It is reasonable to suppose that using thin sections of up to 13 mm with fibres of length 12 mm, is more likely to lead to the manufacture of specimens with most fibres orientated in the tensile axis direction.

2. Process 2 (where the water is added before the fibres) is preferred for enhancing the workability of the ECC material in its fresh state. Fibre type 1 when used with Process 2 enables enhanced mechanical performance (a stress up to 5 MPa associated with a strain from initial crack of more than 2.0%). The casting method would play an important role in the mechanical performance.

3. A better alignment of the fibres along the tensile axis would explain the enhanced mechanical performance of the specimens cast in a mould compared with the specimens cut from a slab. Specimen thickness seems to control the degree of contribution of the fibres to the mechanical performance and understanding this contribution is crucial for the design of the ECC material for a particular application. The present work has quantified the fibre orientation distribution and linked it to mechanical performance.

4. Fractographic analysis of specimens tested in tension revealed signs of detached PVA material on the pulled-out fibres, showing a certain resistance offered by the composite fibre-cement to the tensile stress. This demonstrates the importance of the fibre-cement interface in the pseudo-ductility of the ECC material.
There are a number of areas where further work is required. In particular it would be useful to investigate whether thicker sections might demonstrate an improved performance with a higher fibre volume fraction ($V_f$) and if better control of the manufacturing process leads to greater alignment of the fibres in preferred orientations. In considering both these factors, there is a balance to be met between mechanical performance and economics. In addition, there is a need for a better understanding of the chemistry of the fibre-matrix interface and its performance in aggressive environments.

**Acknowledgements**

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**References**

Fig. 1 Typical tensile stress-strain curve for ECC after [3]
Fig. 2 Schematic of unconfined tensile test based on the Japanese Standard after [10].

Fig. 3 Tensile test arrangement for (a) Original standard dog-bone geometry following the Japanese Standard and (b) Gripping Arrangement.
Fig. 4 (a) Tensile testing results associated with the initial dog-bone geometry and (b) photograph of specimen C2928 exhibiting cracks at the sharp edges
Fig. 5: Tensile test arrangement for (a) Revised dog-bone geometry with a radius of curvature R of 185 mm following the FEA analyses and (b) Gripping Arrangement

Fig. 6: Stress-strain results for specimens tested in tension: effect of fibre type (Process 1)
Fig. 7 Stress-strain results for specimens tested in tension: effect of process type (Fibre type 1)
Fig. 8 Photographs of dog-bone ECC specimens made from the same batch of material following testing (a) sample reference C6541, $\varepsilon = 1.04$ (b) C6542, $\varepsilon = 0.33$ and (c) C6543, $\varepsilon = 2.24$; the quantity $\varepsilon$ represents the strain to failure after the first crack (load-drop) occurs. The arrows indicate clusters of cracks, each of which correlates with one or more load-drop events on the load-displacement (stress-strain) response, see Fig. 6.
Fig. 9 Stress-strain results for specimens tested in tension: comparison of specimens cast in moulds with specimens cast in a slab.

Fig. 10 Tensile failure strain and fracture stress as a function of ECC sample thickness. Data for 13 mm, 20 mm and 60 mm thick samples taken as best values from references [3], [11] and [12]. Data for 30 mm the highest values from the present work.
Fig. 11 Secondary scanning electrons photomicrographs (SEM) from a fractured ECC specimen (C6543) (a) low magnification image showing a number of pulled-out fibres, (b)-(d) higher magnification of single fibres showing different interface and fracture behaviour (20 kV)
Fig. 12 Backscattered scanning electron photomicrograph (SEM) of a polished surface from an ECC sample (C6544C) showing typical porosity and a range of fibre sections corresponding to different orientations.
Fig. 13 (a) Fibre orientation data for three specimens (left to right): C6523, C6543 and C6544C and (b) stress-strain results for these specimens
## Tables

### Table 1: Comparison between Fibre Reinforced Cements (FRC), High Performance Fibre Reinforced Cements (HPFRC) and ECC materials [2]

<table>
<thead>
<tr>
<th></th>
<th>FRC</th>
<th>Common HPFRC</th>
<th>ECC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composite Design Methodology</td>
<td>N/A</td>
<td>Use high Vf</td>
<td>Micromechanics based, minimise Vf for cost and processability</td>
</tr>
<tr>
<td>Fibre</td>
<td>Any type, Vf usually &lt; 2%; d_f steel ≤ 500 µm</td>
<td>Mostly steel, Vf usually &gt; 5%, df = 150 µm</td>
<td>Tailored, polymer fibres most suitable; Vf usually &lt; 2%; d_f &lt; 50 µm</td>
</tr>
<tr>
<td>Matrix</td>
<td>Coarse aggregates used</td>
<td>Fine aggregates used</td>
<td>Controlled for matrix toughness and initial flaw size; fine sand used</td>
</tr>
<tr>
<td>Interface</td>
<td>Not controlled</td>
<td>Not controlled</td>
<td>G_c and f_c controlled</td>
</tr>
<tr>
<td>Tensile behaviour</td>
<td>Strain-softening</td>
<td>Strain-hardening</td>
<td>Strain-hardening</td>
</tr>
<tr>
<td>Tensile strain capacity</td>
<td>0.10%</td>
<td>&lt; 1.5%</td>
<td>&gt; 3% ; 8% demonstrated</td>
</tr>
<tr>
<td>Crack width</td>
<td>Unlimited</td>
<td>Typically several hundred µm, unlimited for ε &gt; 1.5%</td>
<td>Typically &lt; 100 µm during strain-hardening</td>
</tr>
<tr>
<td>Processing</td>
<td>Self-compaction demonstrated; Extrudability demonstrated</td>
<td>Self-compaction impossible due to high Vf, often requires high frequency vibration (e.g. in CRC); Extrudability demonstrated</td>
<td>Self-compaction demonstrated; Extrudability demonstrated</td>
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### Table 2: Details of the ECC mix design

<table>
<thead>
<tr>
<th>Constituent details</th>
<th>Content</th>
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<tr>
<td>Portland cement (c)</td>
<td>W/c = 0.68</td>
</tr>
<tr>
<td>Fine aggregates (a)</td>
<td>a/c = 0.68</td>
</tr>
<tr>
<td>Water (w)</td>
<td>w/c = 0.34</td>
</tr>
<tr>
<td>Admixtures (ad)</td>
<td>ad/c = 0.006</td>
</tr>
<tr>
<td>Fibres (f)</td>
<td>2 % Vol.</td>
</tr>
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</table>

### Table 3: Experimental test matrix relating to fibre and process types (LD = Cross-head displacement rate during testing)

<table>
<thead>
<tr>
<th>Type Fibre Type</th>
<th>Process 1</th>
<th>Process 2</th>
</tr>
</thead>
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<tr>
<td>Type 1</td>
<td>LD = 0.05 mm/min</td>
<td>LD = 0.05 mm/min</td>
</tr>
<tr>
<td>Type 2</td>
<td>LD = 0.05 mm/min</td>
<td>N/A</td>
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</table>
### Table 4 Mechanical and Physical Property data for various test specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fibre and Process</th>
<th>Workability</th>
<th>Casting</th>
<th>Age tested (days)</th>
<th>$\sigma_{\text{mean,EC}}$ (MPa)</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$\varepsilon_{\text{from,EC}}$ (%)</th>
<th>Density (kg/m$^3$)</th>
<th>Porosity (%)</th>
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<tbody>
<tr>
<td>C6256</td>
<td>T2/P1</td>
<td>175 mm</td>
<td>mould</td>
<td>27</td>
<td>4.3</td>
<td>4.3</td>
<td>0.37</td>
<td>1951</td>
<td>4.6</td>
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<td>C6257</td>
<td>T2/P1</td>
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<td>mould</td>
<td>27</td>
<td>4.3</td>
<td>4.4</td>
<td>0.95</td>
<td>1940</td>
<td>4.9</td>
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<td>C6258</td>
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<td>mould</td>
<td>27</td>
<td>3.7</td>
<td>3.9</td>
<td>0.53</td>
<td>1946</td>
<td>5.1</td>
</tr>
<tr>
<td>C6522</td>
<td>T1/P2</td>
<td>208.5 mm</td>
<td>mould</td>
<td>27</td>
<td>4.3</td>
<td>4.3</td>
<td>0.30</td>
<td>1969</td>
<td>5.0</td>
</tr>
<tr>
<td>C6523</td>
<td>T1/P2</td>
<td>208.5 mm</td>
<td>mould</td>
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<td>4.9</td>
<td>2.17</td>
<td>1969</td>
<td>7.8</td>
</tr>
<tr>
<td>C6524</td>
<td>T1/P1</td>
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<td>mould</td>
<td>27</td>
<td>3.9</td>
<td>4.7</td>
<td>1.41</td>
<td>1987</td>
<td>5.4</td>
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<tr>
<td>C6541</td>
<td>T1/P1</td>
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<td>mould</td>
<td>27</td>
<td>3.9</td>
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<td>1.04</td>
<td>1937</td>
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</tr>
<tr>
<td>C6542</td>
<td>T1/P1</td>
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<td>mould</td>
<td>27</td>
<td>3.7</td>
<td>3.8</td>
<td>0.33</td>
<td>1962</td>
<td>4.6</td>
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<tr>
<td>C6543</td>
<td>T1/P1</td>
<td>175 mm</td>
<td>mould</td>
<td>27</td>
<td>3.3</td>
<td>3.9</td>
<td>2.24</td>
<td>1952</td>
<td>9.5</td>
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<tr>
<td>C6544A</td>
<td>T1/P1</td>
<td>172 mm</td>
<td>slab</td>
<td>27</td>
<td>2.4</td>
<td>2.5</td>
<td>0.28</td>
<td>1915</td>
<td>7.8</td>
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<tr>
<td>C6544B</td>
<td>T1/P1</td>
<td>172 mm</td>
<td>slab</td>
<td>27</td>
<td>2.2</td>
<td>2.2</td>
<td>0.16</td>
<td>1913</td>
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<tr>
<td>C6544C</td>
<td>T1/P1</td>
<td>172 mm</td>
<td>slab</td>
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<td>2.3</td>
<td>2.3</td>
<td>0.13</td>
<td>1925</td>
<td>7.1</td>
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### Table 5 Polishing and grinding stages

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<th>Stages</th>
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<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
<th>VII</th>
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<tr>
<td>Equipment</td>
<td>Planopol</td>
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<tr>
<td>Specimen holder</td>
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<tr>
<td>Grinding media</td>
<td>SiC</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Grit/Grain size</td>
<td>81 µm</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
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<tr>
<td>Polishing cloth</td>
<td>/</td>
<td>Metal bond</td>
<td>Resin bond</td>
<td>DP-DUR (silk cloth)</td>
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<td></td>
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<tr>
<td>Polishing media</td>
<td>/</td>
<td>Diamond pad</td>
<td>Diamond Suspension</td>
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<tr>
<td>Grain (µm)</td>
<td>/</td>
<td>75</td>
<td>20</td>
<td>10</td>
<td>6</td>
<td>3</td>
<td>1</td>
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<tr>
<td>Lubricant</td>
<td>WATER</td>
<td>BLUE</td>
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<tr>
<td>Speed (rpm)</td>
<td>300</td>
<td>150</td>
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<td></td>
<td></td>
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<td>Time (mins)</td>
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<td>10</td>
<td>5</td>
<td>2</td>
<td>2</td>
<td>2.5</td>
<td>3</td>
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### Table 6 Fibre orientation and dispersion data for ECC samples based on T1 fibres taken from moulds (prepared by two different manufacturing routes: specimens C6543 and C6523) and a slab (specimen: C6544C) (SI)

<table>
<thead>
<tr>
<th>Angle</th>
<th>$\theta$</th>
<th>$\theta_{\text{rad}}$</th>
<th>Nbr (%)</th>
<th>$V_\text{s}$</th>
<th>$\eta_\text{s}$</th>
<th>Nbr (%)</th>
<th>$V_\text{s}$</th>
<th>$\eta_\text{s}$</th>
<th>Nbr (%)</th>
<th>$V_\text{s}$</th>
<th>$\eta_\text{s}$</th>
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</thead>
<tbody>
<tr>
<td>0 - 20°</td>
<td>10</td>
<td>0.17</td>
<td>2073</td>
<td>15.95</td>
<td>0.16</td>
<td>0.150</td>
<td>812</td>
<td>6.88</td>
<td>0.07</td>
<td>0.065</td>
<td>592</td>
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<tr>
<td>20 - 30°</td>
<td>25</td>
<td>0.44</td>
<td>4216</td>
<td>32.44</td>
<td>0.32</td>
<td>0.219</td>
<td>2380</td>
<td>20.17</td>
<td>0.2</td>
<td>0.136</td>
<td>1827</td>
</tr>
<tr>
<td>30 - 40°</td>
<td>35</td>
<td>0.61</td>
<td>2864</td>
<td>22.03</td>
<td>0.22</td>
<td>0.099</td>
<td>2755</td>
<td>23.53</td>
<td>0.23</td>
<td>0.105</td>
<td>2719</td>
</tr>
<tr>
<td>40 - 50°</td>
<td>45</td>
<td>0.79</td>
<td>1410</td>
<td>10.85</td>
<td>0.11</td>
<td>0.027</td>
<td>2168</td>
<td>18.37</td>
<td>0.18</td>
<td>0.046</td>
<td>2499</td>
</tr>
<tr>
<td>50 - 60°</td>
<td>55</td>
<td>0.96</td>
<td>925</td>
<td>7.12</td>
<td>0.07</td>
<td>0.008</td>
<td>1662</td>
<td>14.08</td>
<td>0.14</td>
<td>0.015</td>
<td>2163</td>
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<td>60 - 70°</td>
<td>65</td>
<td>1.13</td>
<td>1076</td>
<td>8.28</td>
<td>0.08</td>
<td>0.003</td>
<td>1367</td>
<td>11.58</td>
<td>0.12</td>
<td>0.004</td>
<td>1754</td>
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<tr>
<td>70 - 90°</td>
<td>80</td>
<td>1.4</td>
<td>434</td>
<td>3.34</td>
<td>0.03</td>
<td>3.00E-05</td>
<td>656</td>
<td>5.56</td>
<td>0.06</td>
<td>5.00E-05</td>
<td>1236</td>
</tr>
<tr>
<td>Total</td>
<td>12998</td>
<td>100</td>
<td>1</td>
<td>0.506</td>
<td>11800</td>
<td>100</td>
<td>1</td>
<td>0.371</td>
<td>12790</td>
<td>100</td>
<td>1</td>
</tr>
</tbody>
</table>

| $\eta_\text{s}$ | 1 |
| $\eta_\text{a}$ | 0.51 | 0.37 | 0.31 |
| $\sigma_{\text{max}}$ (MPa) | 15.8 | 11.6 | 9.6 |