Manufacturing Processes for Engineered Cement-Based Composite Material Products

BY

DON DE KOKER

Thesis presented in partial fulfilment of the requirements for the degree of Master of Engineering (Civil) at the University of Stellenbosch.

Study Leader: Prof G.P.AG. van Zijl.

December 2004

Declaration:

I, the undersigned, hereby declare that the work contained in this thesis is my own original work and that I have not previously in its entirety or in part submitted it at any university for a degree.

Signature: ……………………………….

Date: ………………………………….

Synopsis:

The effort to modify the brittle behaviour of plain cement materials such as cement pastes, mortars and concretes has resulted in the modern concepts of fibre reinforcement and matrix-fibre interface engineering. The behaviour of such modern cement-based materials is characterized by a more ductile post-peak softening in uni-axial tension compared with the plain, unreinforced matrix. This is as a result of balancing the matrix strength and toughness with fibre bond and strength. The mechanical response shows a sustained or even higher tensile load carrying capacity after first cracking of the matrix. This class of fibre reinforced composites, designed to exhibit such pseudo-strain hardening properties based on micromechanical principles, are referred to as engineered cement-based composites (ECC).

ECC are manufactured by either cast moulding, extrusion or spinning (in the case of pipes). Different manufacturing techniques lead to different performance of composites.
Researching the micro-mechanical aspects of ECC, particularly fibre orientation and fibre-matrix interfacial bond strength, leads to a better understanding of the strength characteristics and mechanical behaviour. Moreover, the manufacturing process significantly influences these characteristics. Here lies the focus of this research.

The research consisted of an in-depth research, literature study and understanding of ECC technology, developing extrusion and cast equipment, developing laboratory testing equipment for pipe testing, tailoring the ECC mix for the specific purpose of manufacture, manufacturing of ECC plate and pipe samples, testing of ECC for various manufacturing techniques and scrutinising/ analysing the results for recommendations with respect to further research and commercialisation of ECC material products.

Sinopsis:

Beton, mortel en sement produkte se bros gedrag onder belasting het nuwe konsepte in vesel-versterkte bewapening tot gevolg gehad. Veselversterkte beton se gedrag word gekarakteriseer deur die verhoogde weerstand in trek na aanvanklike faling. Deur die matrikssterkte en die veselkarakteristieke te balanseer, kan veelvuldige krake veroorsaak word, met gepaardgaande geleidelike weerstandsverhoging. Hierdie klas van veselversterkte sement produkte word na verwys as 'Engineered Cement-based Composites' (ECC).

ECC produkte kan vervaardig word deur die produk te giet, te ekstrueer of in die geval van pype, te spin. Die vervaardigingsproses het 'n noemenswaardige invloed op die sterkte-gedrag van die ECC.
Ondersoek na die mikro-meganiese gedrag van ECC, hoofsaaklik veseloriëntasie en die vesel-matriks interaksie, is nodig om die sterkte-gedrag van ECC te verstaan en verder te ontwikkel binne die raamwerk van die spesifieke vervaadigingsmetodes. Hier lê die fokus van hierdie studie.

Die navorsing het die volgende behels: deeglike literatuurstudie, die ontwikkeling van apparatuur vir ekstrusie en giet, die ontwikkeling van laboratorium toetsapparatuur (vir pype), ECC mengontwerp vir die spesifieke vervaardigingstegnieke, vervaadiging van ECC plaat-monsters en pyp-monsters, die toets daarvan en interpretasie van resultate met betrekking tot die maak van aanbevelings ten opsigte van verdere navorsing en kommersialisering van ontwikkelde tegnologie.

Acknowledgements:

I wish to thank my study leader, **Prof. Gideon van Zijl**. His assistance, systematic approach to all problem-solving and great knowledge contributed tremendously to my understanding of concepts and approach to problems. He was the co-ordinator of all my research. Gideon is the most supportive lecturer I ever came across.

I would like to express my gratitude to **Mr Andries Rossouw** from the workshop. He is very innovative and practical. His input and effort is much appreciated.

Mr Billy Boshoff and Mr Arthur Layman supplied high quality assistance in the laboratory**.** Thank you Billy for your over-all support, your input on mix characterisation and your organising of events in the laboratory.

We acknowledge the support of **INFRASET** Infrastructure Products, especially Mr J. Kleynhans, for their tremendous support and financial backing.

We also acknowledge the support of **University Pretoria** for sharing their laboratory and scanning electron microscope (SEM) facilities, as well as their helpful discussions regarding paste characterisation. We make special exception in thanking **Mr Derik Mostert** and **Prof. Elsabé Kearsley**.

Thank you **Mrs M. Lotter** for seeing to the financial and organisational (i.e. travel arrangements, etc.) aspects.

The supply of fibre materials by **Kuraray Co. Ltd.**
The supply of viscous agent (VA) and super-plasticizers (SP) by **Chryso**.

The support of this research by the **Cement and Concrete Institute (C&CI)**, as well as the **Technology and Human Resources in Industry Programme (THRIP**) of the South African Ministry of Trade and Industry is gratefully acknowledged.

Everyone else who I was not able to mention, but who also contributed to this thesis.
Thank you.

Table of Contents:

List of Figures:

List of Tables:

Nomenclature:

List of Acronyms:

CT Scan	Computed Tomography Scan
ECC	Engineered Cement-based Composites
EDS	Energy Dispersive Spectrometry
EECC	Extruded Engineered Cement-based Composites
FA	Fly Ash
FM	Fineness Modulus
FRC	Fibre Reinforced Concrete
GGBFS	Ground Granular Blast Furnace Slagment
GGCS	Ground Granular Corex Slagment
HFC	Hybrid Fibre Concretes
LVDT	Linear Variable Displacement Transducer
MOR	Modulus of Rupture
NMR	Nuclear Magnetic Resonance
PVA	Polyvinyl Alcohol
SABS	South African Buro of Standards
SEM	Scanning Electron Microscope
SF	Silica Fume
SFRC	Steel Fibre Reinforced Composites
SFR-ECC	Steel Fibre Reinforced Engineered Cement Based Composite
SP	Super Plasticizer
VA	Viscous Agent (Methyl Cellulose)

List of Symbols:

σ_u	Ultimate compressive strength (of composite)
σ_{tu}	Ultimate tensile strength (of composite)
σ_f	Fibre tensile strength
E	Young's modulus
ε_f	Ultimate tensile strain
β	Fibre bond factor
V_f	Fibre volume fraction
g	Fibre snubbing factor
τ	Fibre bond factor
F	Fibre factor
θ	Angle between the transition section wall and the direction of extrusion; slope of the transition section
θ_c	Maximum angle θ at which ECC can still be pushed forward under a horizontal force
η	Surface frictional resistance

1. Introduction

Engineered cement-based composites (ECC) have superior mechanical properties in tension. Through engineering tailored ingredient proportions, this class of materials exhibits tough behaviour in tension, as opposed to brittle behaviour of normal concrete.

Like in fibre reinforced concrete and cements (FRC), the fibres perform crack bridging in the matrix. Thereby, the fibres arrest and divert micro-cracks developing in the matrix. The superiority of ECC to FRC lies in the strain hardening or increased tensile resistance beyond the initial cracking strain. Through sufficient crack bridging, a next weak point in the matrix is brought to its limit strength; leading eventually to multiple, fine cracks, as opposed to a single localization crack in FRC. Through micro-mechanics based modelling, it has been shown that material and geometrical properties of the fibres, as well as stiffness and strength properties of the matrix and the fibre-matrix interaction determine the multiple cracking, pseudo-hardening tensile response of ECC (Li et al., 1995). The performance of ECC's depends largely on the properties of fibres and matrix, and the characteristics of the fibre-matrix interface. Furthermore, in the post-peak region of the tensile stress-strain behaviour, the number of fibres per unit area of the cracked section plays a governing role. Thus, apart from the fibre and matrix properties, the fibre dispersion, as well as orientation, plays an important role.

Good matrix properties and fibre-matrix interaction are achieved by decreasing the amount and size of aggregate, which tend to adversely affect the ductile behaviour of the composite. Another prerequisite for strain hardening is balancing the matrix and fibre properties for effective crack bridging. Thereby, unlike some high performance FRC, ECC does not utilize large amounts of fibres.

In addition to matrix-fibre tailoring, an improvement in ECC behaviour can be brought about by favourable orientation of fibres. Therefore, to optimise ECC and exploit its superior characteristics commercially, it should be tailored for industrial fabrication processes, whereby the influence of the particular processes can be employed to achieve the desired effects. The processes influence the material directly, through fibre orientation and modification of fibre-matrix interfacial properties, as well as indirectly, by the required adjustments to the mix ingredients and proportions.

This study addresses the issues outlined above. Standard casting and vibration and extrusion processing of ECC are discussed. Required mix adjustments for these processes are elaborated, illuminating the different mix designs for optimal performance of products for the different processes. Evidence of production specific orientation of fibres is presented. The influence of the processes on the mechanical properties is discussed at the hand of results of standard laboratory tests.

Extrusion is a plastic-forming method whereby several structural shapes, such as pipes, are manufactured. The manufacturing process includes premixing, extrusion and curing. The introduction of extrusion moulding in cement product processing entails the formation of cement products under high shear and high compressive forces. This leads to performance-enhancing densification of the material, as well as the potential of producing products of superior geometrical tolerance. Furthermore, it has a beneficial influence on the fibre orientation.

There is evidence that short fibres are aligned by extrusion, leading to significantly improve mechanical properties of the material. Fibre alignment manifests in the main direction of the extrusion mechanism. Consequently, the mechanical properties of the fibre-reinforced composite are improved in that direction.

The research studies the potential improvement to ECC material and product behaviour by extrusion, while the influence of other, more traditional processes of manufacturing are studied as reference. To perform the research and comparison, optimal mix design for extrusion is sought. Properties for extrudability include fluidity and viscosity through moisture content, viscosity agent and aggregate grading, fibre type, fibre content and fibre aspect ratio. The extrusion equipment was manufactured, from which test results were generated. Finally, a reference test matrix was prepared to clarify the influence of these parameters on product geometry and mechanical behaviour in tension and bending.

In the light of the above description of ECC material characterisation, design and improvement, the particular objectives of this research project are stated next.

1.1 Primary research objective:

Quantification of fibre orientation and matrix densification are part of a more detailed endeavour to study and improve the mechanical behaviour of ECC by manufacturing technique. Though quantification of fibre orientation is not achieved in this research, an investigation into the effects of the manufacturing technique on fibre orientation and the methods of determining the fibre orientation is studied. This investigation is set as the goal for the current study in order to substantiate the effectiveness of the manufacturing process with respect to fibre orientation and matrix densification. With the aid of the CT scan method, coupled with the use of steel fibres as the fibre constituent, images are obtained from which fibres are distinguished from the rest of the matrix. The difference in densities between the fibre and the rest of the matrix is a requirement for obtaining clear CT scan images, which explain the reason for using steel fibres as the fibre constituent. Evidence of production specific fibre orientation and densification is sought. Standard casting and vibration, pipe spinning, piston extrusion processing and auger extrusion processing of ECC are the processes being investigated and compared.

In addition to the fibre orientation investigation, evidence of its influence on the mechanical properties of the material is sought by producing and testing tensile and bending specimens, as well as larger products of ECC material, in this case pipes. The relevant chapters are 4, 6, 7 and 8.

1.2 Secondary research objective:

Required mix adjustments for the spinning, casting, piston extrusion and auger extrusion processes are developed, leading to the different mix designs for optimal performance of products from the different processes, while keeping basic constituents constant to enable direct comparison of results.
Another secondary objective was the manufacturing of two extruder machines, a plate piston extruder and a hollow pipe piston extruder. Developing the extrusion process and building the apparatus was initially the aim of this research and even though the study evolved into an in-depth study of the effects of the different manufacturing processes, it was still a requirement for completing the test matrix. The relevant chapters are 4 and 5.

1.3 Scope

The research process is illustrated schematically in Figure 1.

In Chapter 2: *Research Significance and Theory* the relevance of this material in the construction industry nationally and internationally is briefly stated. The theory on fibre orientation and interfacial friction bond strength is also outlined in this chapter.

Chapter 3 elaborates the material constituents and properties, after which Chapter 4 describes the experimental philosophy and mix design.

Chapter 5 constitute the manufacture of two extruder machines: a plate piston extruder and a hollow pipe piston extruder. Cast moulds for plate specimens and hollow pipes were developed, and access arranged to both pipes spinning and auger extruder facilities to complete the full range of laboratory work.

Chapter 6 reports and discusses the results of all the laboratory tests. The influence of the processes on the mechanical properties is discussed at the hand of the results.

Chapters 7 & 8 continues the discussion of results, but specifically with regard to the influence of fibre orientation and interfacial friction bond strength on the mechanical properties for the various types of manufactured composites.

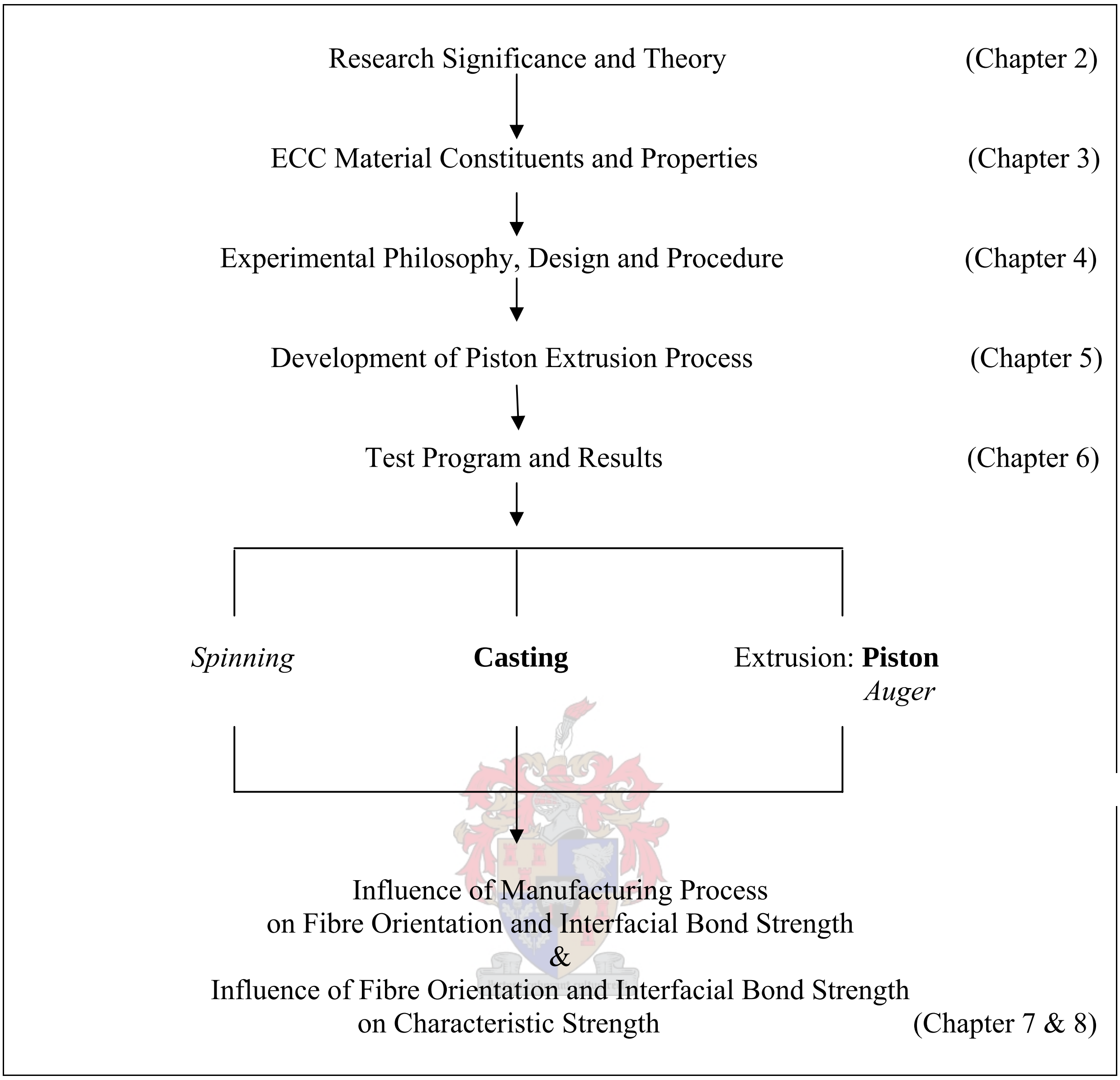


Figure 1: Research proposal diagram

2. Research Significance and Theory

Whereas normal cement-based materials exhibit brittle behaviour, ECC exhibits ductile behaviour in shear and tension. Improved ductility by means of the multiple cracking phenomenon, increased ultimate tensile resistance, the associated reduction in crack width, as well as increased working loads and stresses are the main advantages of ECC products (Boshoff and Van Zijl, 2003). In the post-peak region of the tensile stress-strain behaviour, the number of fibres per unit cross-sectional area of the cracked section plays a governing role – it is an important factor in deciding the peak tensile strength and post-elastic ductility of fibre reinforced cement-based composites. Given a sufficient number of crack-bridging fibres and favourable fibre pullout at the cracked section, multiple cracking is cultivated, characterized by pseudo strain hardening or a sustained increase in load capacity beyond the first matrix cracking resistance. Thus an increase in peak tensile strength and higher post-peak ductility are affected, which distinguish ECC from normal fibre reinforced concrete (FRC).

Applications demand enhanced mechanical performance in the direction of the critical failure mode. Complete exploitation of the benefits of ECC in manufactured products can thus only be obtained when the fibre orientations are aligned with principle directions of rupture. This implies fibre orientation in the longitudinal direction for uni-axial structural elements such as bars, beams or one-way spanning plates, while fibres would have to be aligned orthogonally or perhaps diagonally in bi-axially loaded elements such as two-way spanning plates, or pressure pipes.

The choice of production process can produce the desired orientation. Various processes produce specific fibre orientations: one dimensional alignment of fibre by piston extrusion, two dimensional diagonal fibre alignment of auger extrudate, two dimensional random fibre orientation of thin cast composite, and three dimensional random orientation of thick walled cast composite. For instance, evidence exists of perfect axial alignment in piston-extruded products (Li et al., 2003). Such orientation is best exploited in one-way spanning beams or plates. For thin two-way spanning slabs or plates normal casting may be preferred, whereby fibres are believed to have a random orientation in the plane of the slab. Alternatively, diagonally orientated fibres could be successful in reinforcing products where orthogonal action occurs, such as in pipes and pressure vessels, although such anisotropy may produce undesirable results.

Apart from their particular influence on fibre orientation, the cast and extrusion processes have been chosen because of the broad range in current and potential application for ECC they include. Each method has its benefits, ranging from cost-effective bulk application to the manufacturing of high precision, low porosity products. The properties of the composite are influenced by the method of processing (Delvasto et al. 1986; Igarashi et al. 1996; Shah & Peled 1998; Peled et al. 2000). For instance, extrusion moulding of ECC lowers the porosity of extruded composites by mechanical compaction. Whereas aligned fibre orientation may enhance mechanical properties of the fibre composite in the direction of extrusion, the lower porosity increases the composite strength and matrix toughness (Li et al. 2003).

The study of both fibre orientation and matrix densification will assist in identifying appropriate manufacturing processes for particular applications.

2.1 Fibre orientation

One advantage of using extrusion in cement product processing is that material is formed under high shear and high compressive forces. A further advantage specific to fibre reinforced cement products is that, with properly designed dies and a properly controlled mix, fibres can be aligned in the load-bearing direction. Fibres can be aligned perpendicular to the extrusion direction when extruded with the auger extruder, as opposed to fibre-oriented parallel to the extrusion direction in the case of piston extrusion.

Fibre orientation enhances the mechanical properties of the fibre composite in the direction of the fibre alignment. In doing so, fibres are aligned optimally for resistance of actions in that particular direction. Thereby, larger resistance for the same fibre volume can be achieved or reduced fibre content may be used for a particular required resistance. Furthermore, the ductility of the composite extrudate may be improved by enhancing the multiple cracking phenomenon. Multiple cracking produces pseudo strain hardening characterized by an increase in load capacity after the occurrence of the first crack in the matrix.

In standard cast and vibration applications, fibres orientate randomly, unless influenced by the geometrical boundaries of the mould. Individual fibres can also interact to determine the final orientation, but aggregate particle size grading and distribution probably have the largest influence on the orientation of the fibres.

2.1.1 Fibre orientation determination methods

Observation techniques used to study fibre orientation and dispersion in ECC allows us to calculate fibre orientation. Some techniques are more accurate than other.

2.1.1.1 Optical microscope - (2D method)

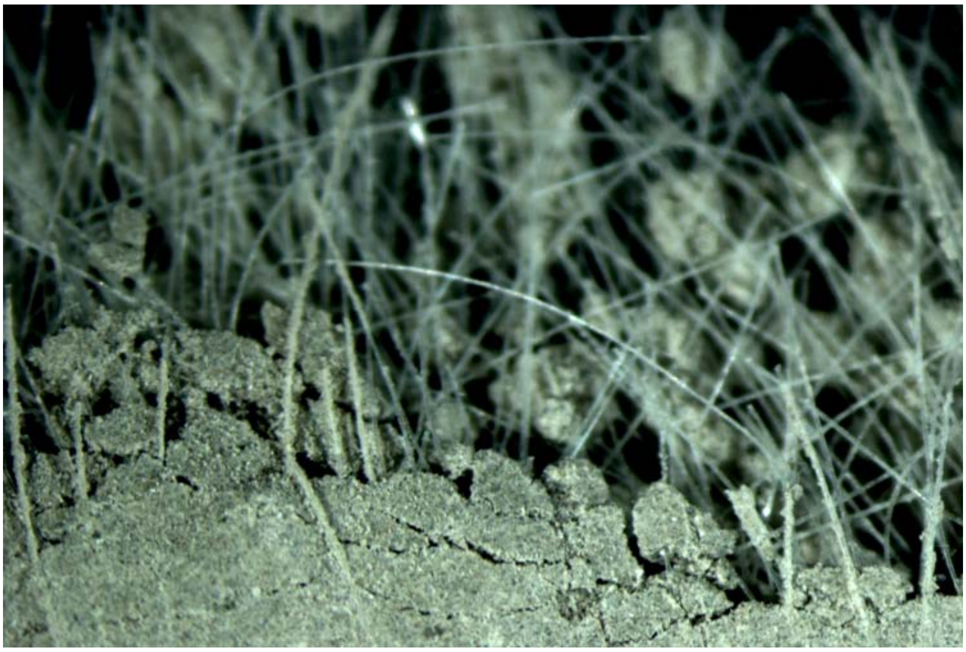


Figure 2: PVA plate extrudate optical microscope image

The optical microscope is easy to use, but restricted to a surface image (2D) of the composite, and furthermore requires the fibres themselves to be exposed or sticking out to determine their alignment as shown in Figure 2. It is unpractical to quantify the fibre orientation with this method.

2.1.1.2 Scanning electron microscope (SEM) and/or energy dispersive spectrometry (EDS) – (2D methods).

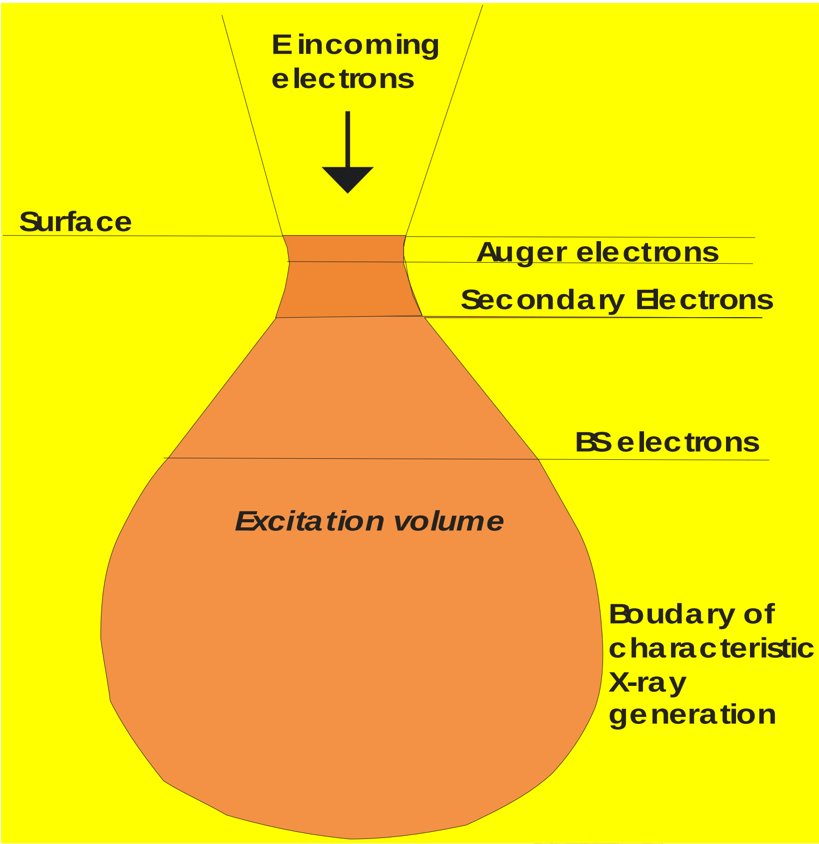


Figure 3: Diagram of scanning electron microscope (SEM) and energy dispersive spectrometry (EDS) analysis material excitation volume

The SEM and EDS analysis are used on samples where the surface preparation doesn't damage the fibre at the surface. The surface is exposed to a high energy electron charge as illustrated in Figure 3. The electrons excite the volume under the surface and generate auger electrons, secondary electrons and backscatter electrons. The backscatter electrons generate images of the characteristic elemental mapping for the specific excitation volume, interpret the feedback and generate visual images of the sample. No interference and a clean sample surface are required.

From the SEM images, the fibre cross-sectional shape and the fibre orientation could be found, as schematized in Figure 4. By assuming a cylindrical shaped fibre, fibre orientation can be estimated by measuring and comparing the perpendicular ordinates of the ellipsoidal cross section, as is also illustrated in the figure.

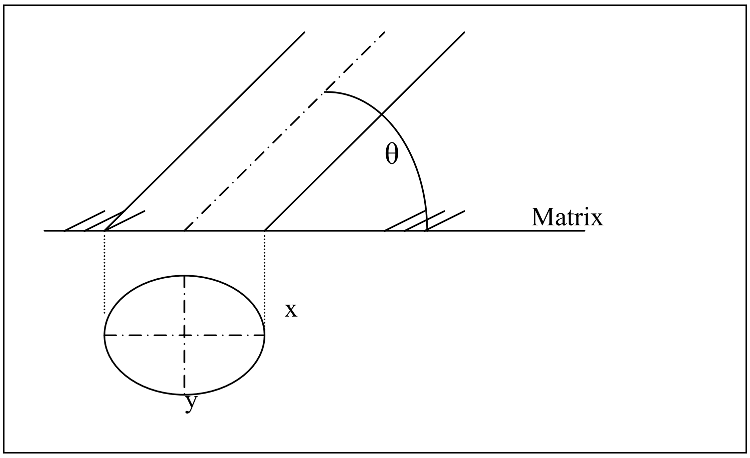


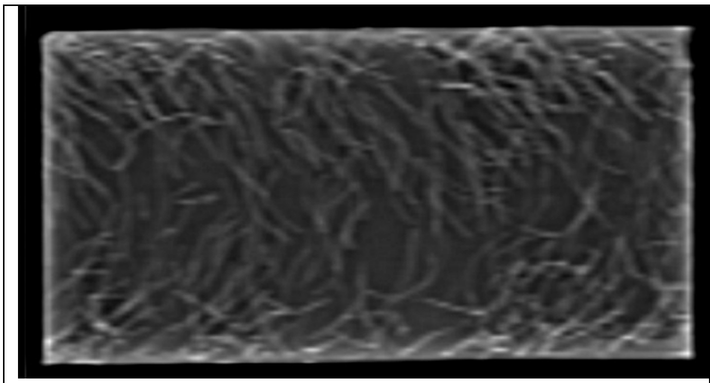
Figure 4: Illustration of fibre orientation - Li et. al. (2003)

2.1.1.3 Computed tomography scan (CT Scan), X-Rays and nuclear magnetic resonance (NMR) – (3D method)

Visualisation of fibres in cement-based composites in a non-destructive way is possible, measured in 3D by micro-focus X-ray computer tomography. In computed tomography scans (CT scans), the intensity of the X-rays is measured over various angles or intervals, and hence enables a 3D visualization of the object.

CT scan images are very useful in determining the fibre orientation since it takes photo-images of the fibres inside the matrix as shown in Figure 5. A requirement for clear CT scan imaging is the difference in densities of the fibre and the rest of the matrix, i.e. steel fibres have to be used to distinguish clearly between the fibre and the matrix.

Nuclear magnetic resonance (NMR) should be used with lower density fibres like PVA. It gives much better resolution, but is very expensive and requires the sample to have no steel constituent present.



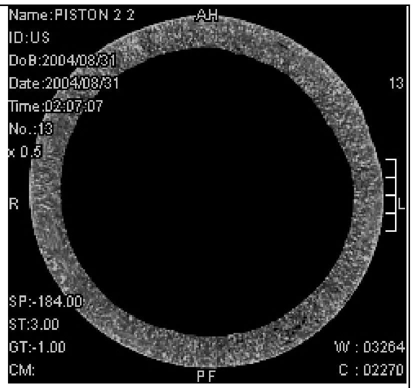


Figure 5 (a): Auger plate extrudate SEM image

Figure 5 (b): Piston extrudate SEM image

Figure 5: Extruded ECC SEM images (showing distinguishable steel fibres)

2.1.2 Factors influencing fibre orientation and distribution

2.1.2.1 Aggregate

The size of the aggregate particles has a significant influence on the distribution of the fibres and the fibre orientation (Figure 6). The fibres in ECC mortar mixes are only separated by fine aggregate particles which are allowed to move freely between the fibres. In conventional FRC, all aggregate particles that are bigger than the average distance between fibres will cause the fibres to become concentrated in balls ("fibre balls") and give rise to irregular distribution of the fibres. This effect will increase in proportion to particle size and has a negative influence on the properties of the concrete. Figure 6 provides an insight into the micro-mechanical phenomenon of improved ductile behaviour for increased amounts of fly ash and fine graded aggregate in ECC. This phenomenon was studied in the paper by Song and Van Zijl (2004), where large proportions of fly ash were introduced and where it acts as well rounded fine aggregate. Coarse aggregates are not used, as they tend to adversely affect the unique ductile behaviour of the composite.

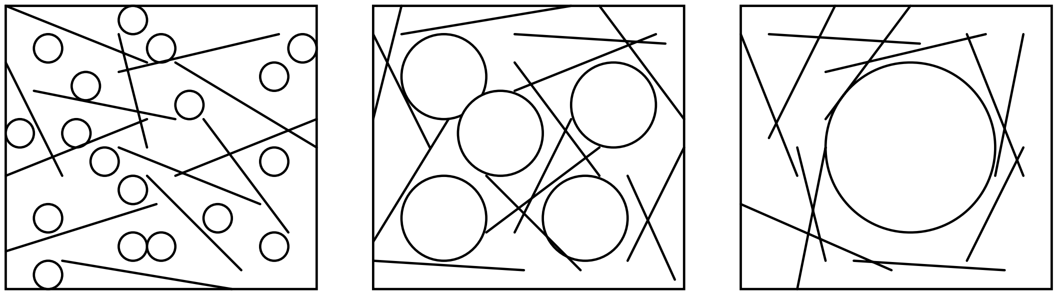


Figure 6(a) Figure 6(b) Figure 6(c)

Figure 6: Particle size influence on fibre distribution, fibre orientation and workability

2.1.2.2 Boundary conditions

When uniformly dispersed in an infinitely large volume of concrete, fibres are expected to be randomly oriented with equal probabilities of being oriented in different directions in space (Figure 7(a)).

Boundaries of the mould or die have an effect on the fibre orientation adjacent to them. In the presence of two parallel boundaries where the distance between the sides are relatively close with respect to the fibre length, the fibres near the boundaries tend to orientate more two-dimensional (2D) near the boundary. Hence, the fibres are in a situation somewhere between three-dimensional (3D) and two-dimensional (2D) random orientations (figure 7(b)).

When there are four closely spaced boundaries (figure 7(c)), there are more restrictions on 3D random orientation of fibres, leading to something between 3D orientation and unidirectional alignment.

Boundaries are present in all manufacturing processes of ECC, and the boundary influence depends on the size of the manufactured composite. It is therefore important to consider this influence (or factor) in deriving theoretical expressions for the orientation factor and the number of fibres per unit cross-sectional area of ECC.

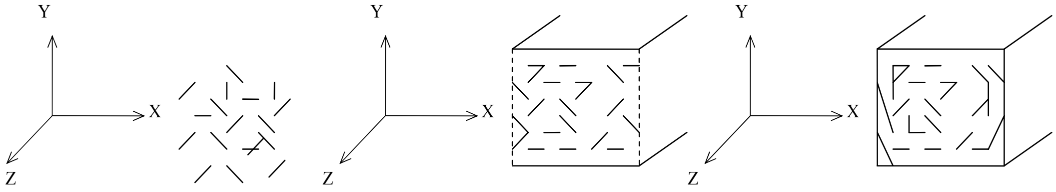


Figure 7 (a) Figure 7 (b) Figure 7 (c)

Figure 7: Illustration of the boundary effect(s) on random orientation of cast composites

2.1.2.3 Vibration

The orientation of steel fibres in ECC (and concrete) and consequently the number of fibres per unit area is not only influenced by the aggregate size and boundaries restricting the random orientations of fibres, but also by the fact that heavy fibres, like steel fibres tend to settle down and reorient in horizontal planes when ECC (and fibrous concrete) is vibrated during placement. As a result of vibration, the orientation of fibres in ECC moves even further away from a 3D fibre orientation condition and tends to approach a 2D condition.

2.1.3 Measurement of fibre distribution

Whereas influences on the fibre orientation are studied in this section, fibre distribution is actually a consequence of fibre orientation, although phenomena like fibre clumping, an extreme case of distribution, do influence the orientation thereof in ECC and SFRC. Fibre distribution runs parallel with fibre orientation in the sense that both distribution and orientation of fibres are affected by the boundaries, aggregate grading and manufacturing process. The number of fibres per unit cross-sectional area is an important factor in the peak tensile resistance and post-peak ductility of the ECC and SFRC (Lee et. al., 1990).

In order to predict the number of fibres per unit cross-sectional area of ECC, the commonly used equation is of the form (Lee et.al., 1990):

$$N_I = \alpha \frac{\mathrm{V_f}}{\mathrm{A_f}} \tag{1}$$

where

N_l = number of fibres per unit area

V_f = volume fraction of steel fibres in concrete

A_f = cross-sectional area of steel fibres

α = orientation factor

2.1.4 Quantifying fibre orientation for ECC products manufacturing

Fibre orientation is generally considered through the use of a so-called fibre orientation factor. Basically, this factor is the average ratio, for all possible fibre orientations, of the projected fibre length in the tensile stress direction to the fibre length itself.

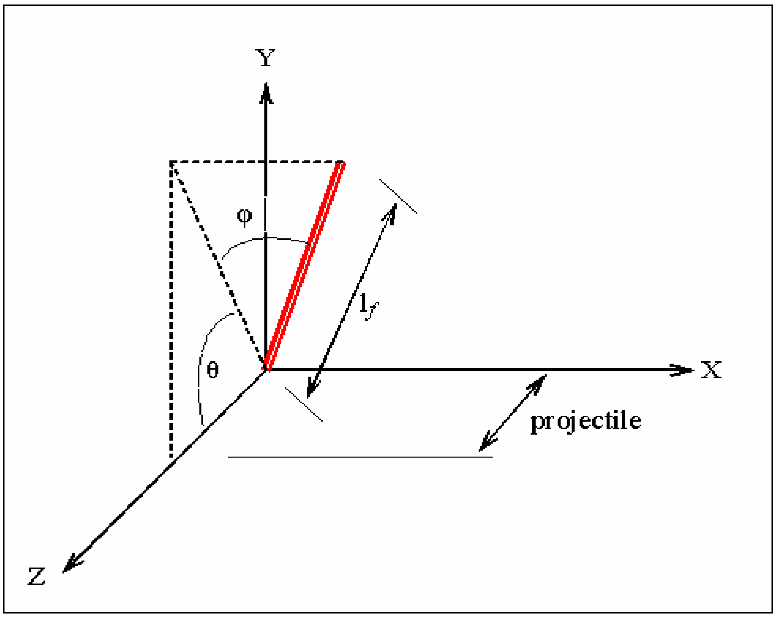


Figure 8: Three-dimensional fibre orientation

2.1.4.1 Quantifying cast composite fibre orientation

In the case of randomly oriented fibres of cast composites like discussed in 2.1.2.2, the orientation factor with the projectile taken along the z-direction (refer Figure 8), can be derived as follows:

$$\alpha_0 = \frac{\int_0^{\pi/2}\int_0^{\pi/2} l_f \cos\theta \cos\phi . d\theta . d\phi}{\left[\frac{\pi}{2}\right]^2 l_f} = 0.405 \qquad (2)$$

where

α_0 = orientation factor

l_f = length of the fibre(s)

cos φ = angle between the fibre and the Y-axis

cos θ = angle between the fibre and the Z-axis

It should be noted that equation (2) is derived for random orientation of fibres in space (Figure 7(a)), where the boundaries don't restrict the freedom of the fibres. Where two or four boundaries are present to restrict the fibre orientation (Figures 7(b) and 7(c)), the orientation factor in the z-direction tends to be greater than the 0.405 derived for 3-D random orientations (Lee et. al., 1990).

2.1.4.2 Quantifying spin and extrudate fibre orientation

The angle between the fibre and the extrusion direction was calculated with the aid of the scalar product or dot product of two vectors. The one vector, say **A**, represents the direction of extrusion, whereas the other vector, say **B**, was calculated for the fibre(s).

The angle between two vectors was then calculated using the following expression:

$$\theta = \cos{-1}\left(\frac{A \bullet B}{|A||B|}\right) \qquad (3)$$

where

A is the vector of any magnitude that represents the extrusion direction

for example 0x + 0y + 1z

B is the vector representing the fibre in space for example 3.7x + 2.9y + 7.3z)

$|A|$ is the scalar of any magnitude representing the extrusion direction given by for example $\sqrt{(0^2 + 0^2 + 1^2} = 1$

$|B|$ is the scalar with magnitude equal to the fibre length for example $\sqrt{3.7^2 + 2.9^2 + 7.3^2} = 13$

The detailed fibre orientation quantification along these lines was not completed in this research for several reasons. For the longer fibres used in auger extrusion, the steel fibres did not stay perfectly straight in the extrusion process, but were bent. This did not happen for the shorter fibre used in piston extrusion and cast products prepared in this research. However, the resolution of the CT scan images makes quantification difficult. These methods should be refined and optimised in future research.

2.2 Fibre-matrix interface bond strength

The performance of ECC's depends largely on the properties of fibres and matrix and the characteristics of the fibre-matrix interface. It is crucial to minimize defects that can occur during the fabrication process, in order to maintain the high strength, fracture toughness, ductility and durability typical of ECC composites. Through extrusion the fibre packing and the matrix is densified whereby the bond between the fibres and the matrix is strengthened. Figure 9 provides a SEM image of the fibre packing and the fibre-matrix interface. It has been argued in the previous section that the performance and properties of fibre cement composites depend on the fibre orientation, which in turn is governed by the method of processing. In this section, the governing role of the fabrication process on the characteristics of the fibre-matrix interface is discussed.

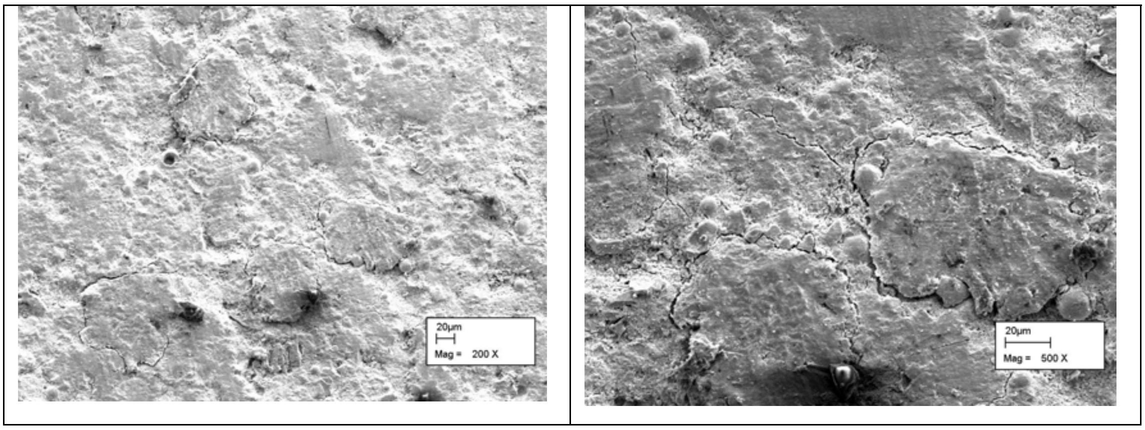


Figure 9: Fibre-matrix interface SEM images

2.2.1 Review of earlier studies

The extent to which the fibres may contribute to the ultimate compressive strength (σ_u) of concrete depends on the fibre aspect ratio (L/d), the volume fraction of fibres (V_f) and the fibre bond factor or frictional shear resistance (τ), enhanced by the snubbing factor (g) formulated by Li, Mishra and Wu (1995) as

$$\sigma_u = ½ . g . \tau . L/d . V_f \qquad (4)$$

Analogous to equation (4) the fibre factor (*F*) has been formulated by Narayanan and Darwish (1990) as

$$F = L/d . V_f . \beta \qquad (5)$$

with β a bond factor accounting for fibre sectional shape, geometrical deformations such as crimped or hooked fibres, or fibre indentations.

The fibre factor (F) is the fibre properties' contribution to the ECC strength and the influence of adjustments to fibre properties on the extrudate can be predicted.

2.2.2 Effect of fibre length

Optimizing the fiber length for cast composites and extrusion leads to improved mechanical performance. It is postulated that successful extrusion, i.e. preventing fibre blockage or inconsistent flow out of the die opening, depends on the ratio of fiber length to die opening. The fibers should be restricted to shorter lengths for a small die opening. Shorter fibers have a more even distribution in the mix and produce increased mechanical behaviour. Longer fibers tend to clump together in the dough-like extrusion mix. Thus, increasing the fiber length is not beneficial for the behaviour of extruded composites, as opposed to enhanced mechanical behaviour of cast composites. Optimization of interfacial bond strength could only be achieved when the fiber length is such that it provides enough resistance to fiber pull-out, yet have a high enough fiber modulus of rupture to avoid fiber fracture.

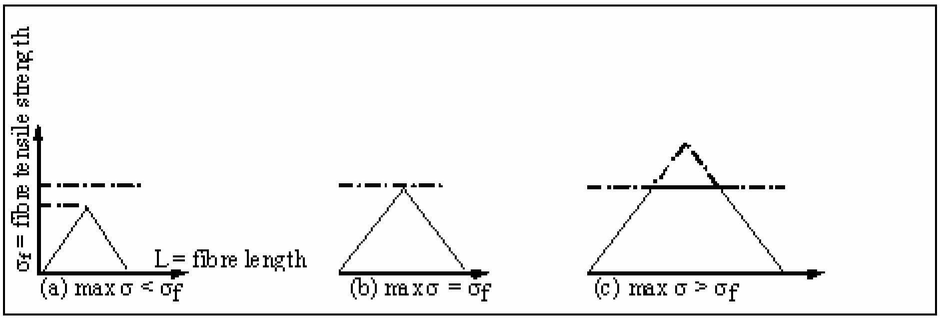


Figure 10: Critical fibre length concept (L_{crit}) – Maidl et. al. (1996)

Failure mode depends on the fibre length (Figure 10). If the fibre length used in the mix design is less than the critical fibre length, fibre pull-out will occur. When the fibre length is longer than the critical fibre length, then fibre fracture is the primary mode of failure.

2.2.3 Anchorage: Fibre Bond Factor (β)

Many of the earlier methods of measuring the bond developed between fibre and cement-based matrices were generally based on simple pull-out tests in which one or both sides of the fibre were embedded in the matrix and the fibre was subjected to direct tension by restraining matrix blocks in compression. Mechanical anchorage of fibres by indentation, crimping or hooding would obviously enhance the bond significantly. We used straight fibres in this research and the bond strength was primarily optimised by fibre orientation and matrix densification.

Earlier studies showed regression analysis derived from these anchorage tests on deformed fibres and conservative bond factor values assigned to different shaped fibres, as: β=0.5 for round fibres, β=0.75 for hooked fibres and β=1.0 for indented fibres.

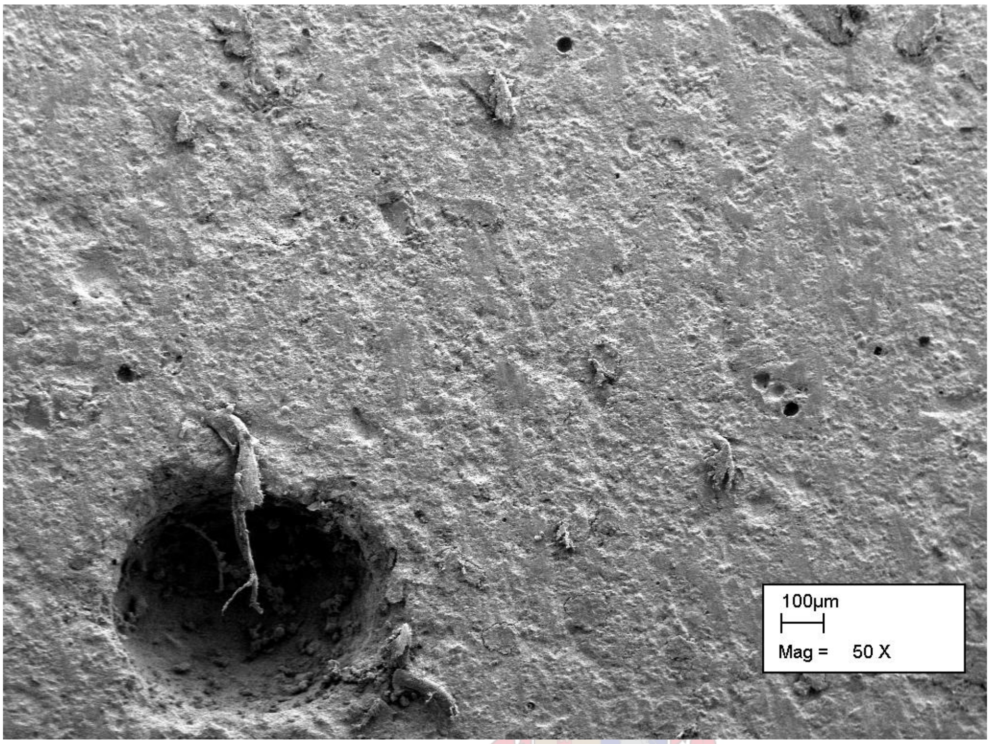


Figure 11: SEM image of fibre pull-out

2.2.4 Aspect Ratio (L/d)

Contradictory behaviour in different fibre geometries for various manufacturing techniques could be explained by the differences in bond strength and matrix properties in different systems, and altering the mode of failure of the fibres until the mechanical performance of the extruded composite is optimized. Extruded composites show more benefit from a lower fibre aspect ratio than what cast composites does. This is due to matrix densification and accompanied increased fibre factor F, causing the tendency of fibre breakage for relatively longer fibres, where longer shear transfer is possible.

Contradicting higher fibre aspect ratios are needed in the case of steel. The steel fibres used in this research has such a high tensile strength that the L/d ratio should be relatively large to optimise the mode of failure, enabling the steel fibres to have better interfacial bond strength, and cultivate higher overall tensile values of the composite.

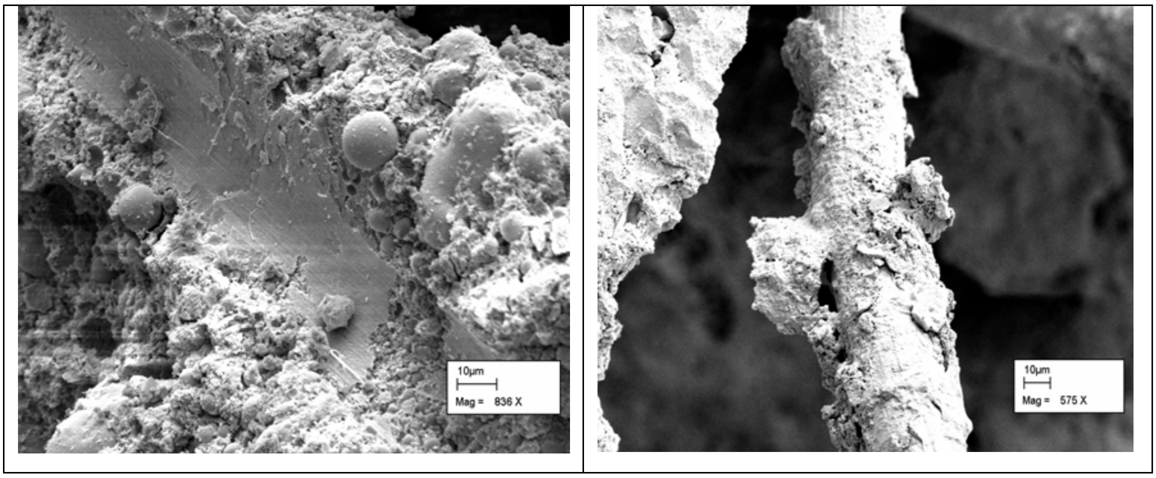


Figure 12: SEM images of ECC showing the matrix-fibre interface

2.2.5 Tensile Strength of Fibres

A requirement to exploit the full interfacial bond potential is that the fibre tensile strength does not limit this resistance. Therefore, it should correspond to whatever ultimate capacity is required from the manufactured composite. This required proportionality between fibre and matrix strength stems from the optimization of the primary mode of failure. Ideally, the probability of fibres pulling out and that of fibre fracture should be equal. Hence, strong matrix properties for high-strength applications require high-strength fibres to withstand the load applied. Hence, higher matrix strength through lower extrudate porosity requires a higher strength fibre.

3. ECC Material Constituents and Properties

In this chapter the ingredients of ECC in general and specifically as used in this research, are briefly described. This is done to introduce the reader to the mix design technology of these materials, as well as to clarify the various influences on the mechanical properties.

3.1 Matrix Constituents

ECC's utilizes the same ingredients as those in FRC, such as water, cement, sand, fibre and chemical additives. Nevertheless, as already shown in Chapter 2, ECC exhibits tensile toughness and pseudo strain hardening after initial cracks arise, as opposed to brittle behaviour of plain concrete and strain softening behaviour of FRC. This phenomenon is illustrated in Figure 13. The superior mechanical response is achieved by, amongst others, decreasing the amount and size of the aggregate, which tend to adversely affect the ductile behaviour of the composite. Also, the matrix strength must be balanced with the fibre type, aspect ratio and fibre volume. Unlike some high performance FRC (Stang et al., 1999), ECC does not utilize large fibre volume. Rather, the combination of ingredients, based on micro-mechanical principles, is what makes the mechanical properties of ECC products so unique.

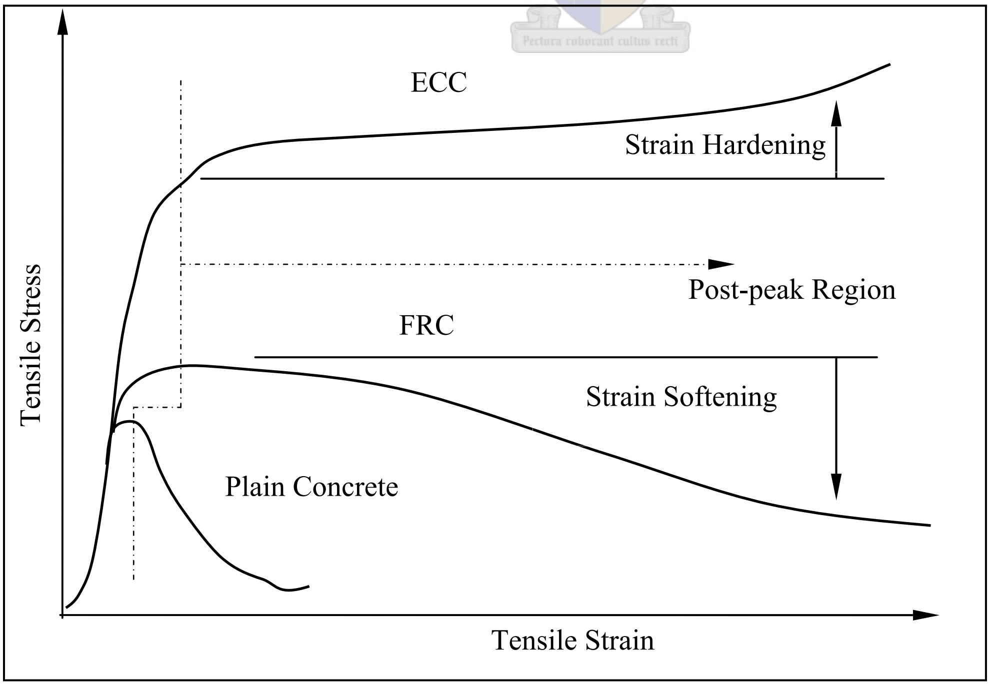


Figure 13: Tensile stress-strain behaviour of cement-based composites

3.1.1 Fibres

Various fibre types like polyvinyl alcohol (PVA) fibres, carbon fibres, polypropylene fibres, micro steel fibres and cellulose fibres, can be incorporated into the cement based matrix.

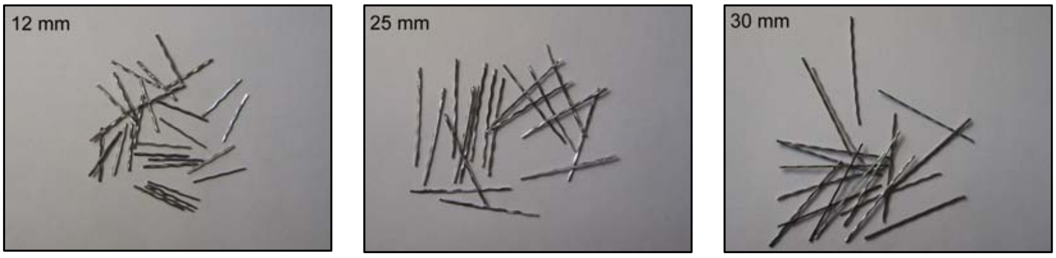


Figure 14: Steel fibres

Figure 15: PVA fibres

The tensile strengths and Young's moduli for various fibres under consideration are shown in Table 1.

Fibre Type	Tensile Strength (GPa)	Elasticity Modulus(GPa)
PVA	0.9-1.6	29-41
Carbon	0.55	45
Polypropylene	0.37	3.6
Micro Steel	1.4-2.3	200
Cellulose	0.5	25-40
Acrylic	0.3	8

Table 1: Fibre properties (Shah et al., 1995 & 2003; Yamada et al., 2004)

In this research, only PVA fibres and micro steel fibres were investigated. The reason for this selection lies in the fact that the tensile strength capacities and stiffness of these fibres are much higher than the other mentioned fibre types. Weaker fibres tend to break in cracked regions, which lead to premature, brittle fracture of the composite. Crack widths are kept small by fibres with high modulus of elasticity.

Research results exist that suggest that fibre length should be greater than 1,5 times the diameter of the largest aggregate particle size in the mix to have a positive influence on the ductility of FRC. The fibres that were used throughout the research programme are listed in Table 2, along with maximum aggregate particle sizes.

	Length (L) (mm)	Maximum diameter (d) (mm)		L/d	Density (kg/m^3)
		Natural	Sieved for this project		
Steel Fibre	13	0,160		81	7,85
PVA Fibre	12	0,040		300	1,3
Aggregate : Philippi		2,36	2,36		2,7
Dolomite Sand		4,75	2		2,7
Mac Sand		4,75	-		2,7
Malmesbury Sand		4,75	-		2,7

Table 2: Fibre geometries and maximum aggregate particle size

From Table 2 it is evident that the fibre lengths used in this research are more than 1,5 times that of the maximum particle size.

3.1.2 Binder

The binder composition comprised of cement (rapid-hardening CEM I - 42,5R for experiments conducted at Pretoria University and Ordinary Portland Cement (CEM I - 42.5) for experiments done at Stellenbosch University), silica fume and fly ash (Ash Resources). The CEM I - 42,5R was used for preparing pipe specimens by spinning, while CEM I - 42.5 was used for all the other specimens. CEM I - 42.5 is hydraulic cement consisting essentially of hydraulic calcium silicates. Since the cement is composed of a heterogeneous mixture of several compounds, the hydration process consists of reactions of the various anhydrous compounds with water occurring simultaneously. As the hydration reaction of cement compounds is exothermic, the compounds of cement are none-equilibrium products of high temperature reactions and are therefore in a high-energy state. The generated heat of hydration could lead to cracks in some cases and affect the structural strength and durability.
With the aid of cement replacement materials such as fly ash and slagment, the heat of hydration could be limited. Fly ash comprises fine particle residue resulting from the combustion of ground or powdered coal (usually in electricity generation plants) and is readily available in the Gauteng province. In some cases cement replacement material in the form of ground Granular Blast Furnace Slagment (GGBFS) are used for this purpose. Saldanha steel manufacturing company in the Western Cape is a source of large volumes of an alternative slagment, namely Ground Granular Corex Slagment (GGCS). Fly ash and GGBFS/GGCS are usually cheap and it is sensible to utilise it as a cost reducing ingredient material for cement-based composite materials. Both fly ash and slagment increase the durability of cement-based composites in the sense that they increase the density of the material and improve the mechanical behaviour, in particular extruded products of ECC. The substantial use of these waste materials from the industry plays an important role in the sustainability in the construction industry. A study was conducted to this effect in parallel with the current research project (Song and Van Zijl, 2004).

The introduction of silica fume (which consists mainly of silica) increases the pozzolanic reaction between cement and the silica where the silica converts the calcium hydroxide into calcium silicate hydrate. Not only does the silica fume play a large role in the pozzolanic reaction, but it also provides dense matrix packing because of its fine particle size.

3.1.3 Chemical Additives

3.1.3.1 Super-plasticizer

The super-plasticizers employed are powerful water reducers. Alternatively, they serve to improve the flow ability, or workability of dry mixes, enabling the use of similar mixes in terms of water and binder content, but which must differ considerately in terms of viscosity and fluidity. Chryso Fluid Premia 100 and Optima 200 are the two types of super-plasticizers used in this research. Both allow the lowest possible water/binder ratios to be used, as required by particular extrusion processes, but retain the easy-to-work aspect of the composite, hence increasing the workability and working time. In addition to reducing the water/binder ratio, its special properties guarantee high short-term strength.

3.1.3.2 Viscous Agent (Methyl Cellulose)

Chryso Aquabeton ZA is a powder additive for concrete for which wash out and segregation of the fresh concrete have to be prevented. For the extrusion process, Aquabeton is an essential additive, since it prohibits the water from being squeezed out under the extrusion pressure. It also serves as a dispersion agent and assists in uniformly dispersing the fibres in the mix not only in extrusion processing, but even in cast and vibration processing. In the latter case, this additive may prevent settling of fibres under gravitational force. The addition of the Chryso Aquabeton ZA will tend to reduce the workability of a concrete or cement-based mix. The starting mix should therefore be designed to take account of this, based on experience from trials. Workability is compensated for with the use of the right water-binder ratio and the right amount of super-plasticizer.

3.1.4 Sand (aggregate)

The important aspect of a cement-based mix is to ensure the distribution of different sand grain sizes. In short we call this spread of grain size the grading of the aggregate. It is important to ensure that the particle sizes are not all the same and that they yield a dense packing of the aggregate, which in turn cultivates workability and densification. Two types of sands were used in our research study. With respect to the research conducted at the University of Pretoria, we used dolomite sand as supplied by Infraset Infrastructure Products, since this was the available sand for Gauteng. Research at Stellenbosch University used mainly the local Philippi Sand in the mix design as supplied by GH Building Supplies, Stellenbosch, South Africa.

3.1.4.1 Dolomite sand

This aggregate has a wide spectrum particle size distribution and in particular, contains a high percentage of fine particles. The smaller particle size ensures optimum matrix particle packing. The fineness modulus (FM) of Dolomite sand is 2,48, where the FM is defined as the method of describing the aggregate by determining the average grain size and given by the following equation:

$$FM = 3{,}31(1+\log D_i) \qquad (6)$$

where

D_i = average grain size in an interval between two sieve sizes measured in mm

The FM of 2,48 indicates that the average particle size for Dolomite sand falls in the interval between 0,3mm and 0,6mm sieves. This sand was used in the spinning process for fabricating pipes. A spinning facility at the Faculty of Civil Engineering, University of Pretoria, was used. Local dolomite sand was used for that exercise.

3.1.4.2. Philippi sand

Philippi sand is finer than Dolomite sand. The FM of Philippi Sand is 1,34 and the average particle size thus falls between 0,15mm and 0,3mm sieve sizes.

3.1.4.3 F70 and F110 sands

In the research leading up to the current study, a large research effort into developing mix designs for ECC was conducted by the research group for cement-based composites at the Civil Engineering Department, University of Stellenbosch. Earlier international research on matrix design for pseudo strain hardening fibre reinforced cement-based composites used graded silica sand conforming to ASTM standards (Li et. al., 1995; Billington and co-workers, 2002). Li et al. (1995) used F50 and F70 fine graded sands (ASTM C 50-70), readily available in the USA, to achieve superior strain hardening behaviour, as well as casting finishes.

In the USA, these grades consist of rounded to sub-angular grains that provide high strength and excellent permeability. The specifications meet the high standards of the U.S. Silica's ISO 9002 Certified Quality System [U.S. Silica Company, Berkeley Springs, West Virginia]. In this research project, sands of these gradings of F70 and F110 were prepared by sieving and proportioning Philippi sand. As this sand lacks the required fine particles, crusher dust was used to complete the required grading.

The grading of sands used in this research are shown in Figure 16 and tabulated in Table 3.

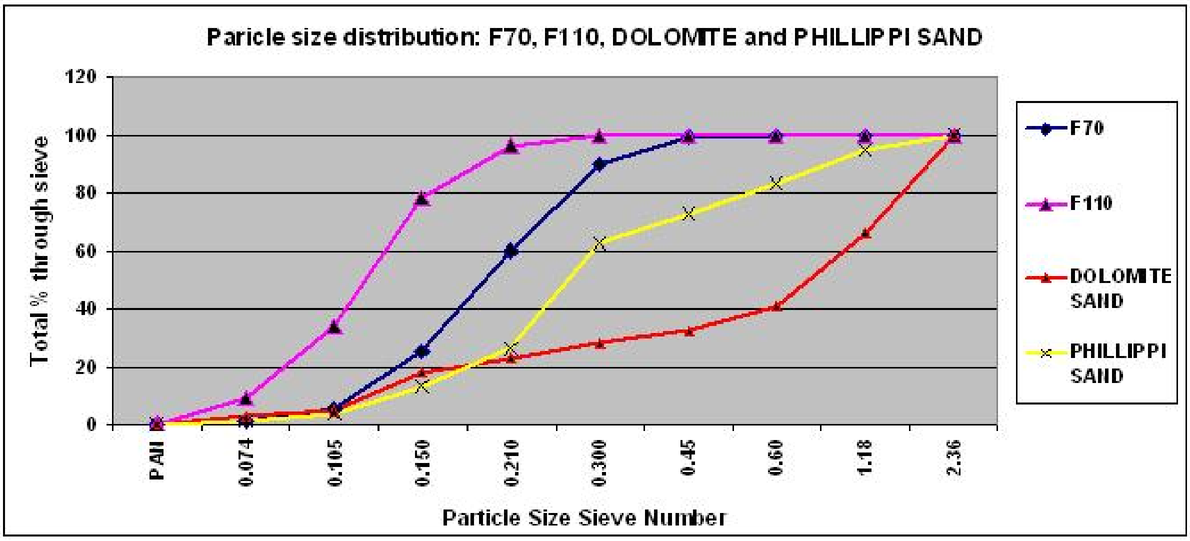


Figure 16: Grading of Dolomite, Philippi, F70 and F110 sands

		Dolomite sand			Phillippi sand		
Particle	Index	Mass on	% on Seive	Total %	Mass on	% on Seive	Total %
Size	Number	Sieve (g)		on sieve	Seive (g)		on sieve
(mm)							
19.00		0	0.00	0.00		0.00	0.00
13.20		0	0.00	0.00		0.00	0.00
9.50		0	0.00	0.00		0.00	0.00
4.75	6	0	0.00	0.00	0	0.00	0.00
2.36	5	0	0.00	0.00	0	0.00	0.00
1.18	4	502	34.10	34.10	29	5.69	5.69
0.60	3	370	25.14	59.24	56	11.14	16.83
0.300	2	190	12.91	72.15	105	20.88	37.70
0.210		74	5.03	77.17	182	36.27	73.97
0.150	1	76	5.16	82.34	64	12.81	86.79
0.100		192	13.04	95.38	49	9.82	96.61
0.074		34	2.31	97.69	13	2.56	99.17
0.053		26	1.77	99.46	2	0.43	99.60
pan		8	0.54	100.00	2	0.40	100.00
	Total	1472	100.00		501	100.00	
FM =				2.48			1.34

Table 3: Sand grading spreadsheet of Dolomite and Philippi sands

		F70 sand			F110 sand		
Particle	Index	Mass on	% on Seive	Total %	Mass on	% on Seive	Total %
Size	Number	Sieve (g)		on sieve	Seive (g)		on sieve
(mm)							
19.00		0	0.00	0.00		0.00	0.00
13.20		0	0.00	0.00		0.00	0.00
9.50		0	0.00	0.00		0.00	0.00
4.75	6	0	0.00	0.00	0	0.00	0.00
2.36	5	0	0.00	0.00	0	0.00	0.00
1.18	4	0	0.00	0.00	0	0.00	0.00
0.60	3	0	0.00	0.00	0	0.00	0.00
0.300	2	100	10.00	10.00	0	0.00	0.00
0.210		300	30.00	40.00	40	4.00	4.00
0.150	1	350	35.00	75.00	180	18.00	22.00
0.100		200	20.00	95.00	440	44.00	66.00
0.074		40	4.00	99.00	250	25.00	91.00
0.053		5	0.50	99.50	45	4.50	95.50
pan		5	0.50	100.00	45	4.50	100.00
	Total	1000	100.00		1000	100.00	
FM =				0.85			0.22

Table 4: Sand grading spreadsheet of F70 and F110 sands

3.2 Mechanical Properties

In cast steel fibre reinforced engineered cement-based composite (SFR-ECC) specimens the fibre parameters which determine ECC toughness and strength include fibre volume fraction, the fibre aspect ratio, length/diameter, the fibre tensile strength and fibre Young's modulus. ECC toughness in this context refers to the area under the stress-strain graph for tensile tests, i.e. a measure of the ability of the specimens to absorb energy during deformation.
With regard to the cement-based matrix the major role players are the matrix tensile strength, Young's modulus, density and, importantly, the aggregate grading.

In the matrix-fibre interfacial zone the potential enhanced composite properties depend on the matrix density, interfacial bond and the orientation of the fibres.

In extruded SFR-ECC products these same parameters remain operational, but the altered realizations of parameters like fibre orientation, as well as the matrix-fibre interfacial zone density, could possibly improve the tensile mechanical behaviour of such products. However, to realize these improvements, successful extrusion of ECC is required, for which the rheology of the batch and the manufacturing parameters must be well controlled to prevent defects such as edge tearing, voids, fibre clogging and other discontinuities. These defects result in reduced performance, because the variability of fibre distribution may be such that low fibre content in critical areas could lead to unacceptable reduction in strength.

Generally, for structural applications, fibres should be used in a role supplementary to reinforcing bars. Fibres can reliably inhibit cracking and improve resistance to material deterioration as a result of fatigue, impact, shrinkage, or thermal stresses. In applications where the presence of continuous reinforcement is not essential to the safety and integrity of the structure, e.g. floors on grade, pavements, overlays, and shotcrete linings, the improvements in flexural strength, impact resistance, and fatigue performance associated with the introduction of fibres in the matrix can be used to reduce section thickness, improve performance, or both.

Fibres influence the mechanical properties of ECC in all failure modes, especially those that induce tensile stress, e.g. direct tension, bending and shear. The strengthening mechanism of the fibres involves transfer of stress from the matrix to the fibre by interfacial shear, or by interlocking between the fibre and matrix if the fibre surface is deformed. Stress is thus shared by the fibre and matrix in tension until the matrix cracks and then the total stress is progressively transferred to the fibres.

Aside from the matrix itself, the most important variables governing the properties of steel fibre reinforced composites are the fibre efficiency and the fibre content. Fibre efficiency is controlled by the resistance of the fibres to pullout, which in turn depends on the bond strength at the fibre-matrix interface. For fibres with uniform section, pullout resistance increases with an increase in fibre length; the longer the fibre the greater its effect in improving the properties of the composite. Also, since pullout resistance is proportional to interfacial surface area, smaller diameter fibres offer more pullout resistance per unit volume than larger diameter fibres because they have more surface area per unit volume. Thus the greater the interfacial surface area or the smaller the diameter, the more effectively the fibres bond. Therefore, for a given fibre length, a high ratio of length to diameter (aspect ratio) is associated with high fibre efficiency. On this basis, it would appear that the fibres should have an aspect ratio high enough to insure that their tensile strength is approached as the composite fails.

An understanding of the mechanical properties of SFR-ECC is an important aspect of successful design. These properties are discussed under the following headings:

- Compression
- Direct tension
- Flexural strength

3.2.1 Compression:

The effect of fibres on the compressive strength of concrete is variable. Documented increases for concrete as opposed to mortar range from negligible in most cases to 23 percent for concrete containing 2 percent by volume of steel fibre, with fibre l/d = 100 and 19mm maximum-size aggregate (Delvasto et. al., 1986). In parallel research in the group at the University of Stellenbosch, compressive strength enhancement by steel fibre of up to 150% has been recorded (Song and Van Zijl, 2005). For mortar mixtures, the reported increase in compressive strength ranges from negligible to slight, although the ductility is nevertheless increased (Delvasto et. al., 1986). For PVA fibre mixes no significant compressive strength increase has been found.

In typical stress-strain curves for SFR-ECC in compression, a substantial increase in the strain at the peak stress can be noted, and the slope of the descending portion is less steep than that of specimens without fibres. This is indicative of substantially higher toughness, where toughness is a measure of ability to absorb energy during deformation, and it can be estimated from the area under the stress-strain curves. The improved toughness in compression imparted by fibres is useful in preventing sudden and explosive failure under static loading, and in absorbing energy under dynamic loading.

3.2.2 Direct tension

The measured tensile values and the observed shape of the stress-strain curve depend on the size of the specimen and whether single or multiple cracking occurs. The ascending part of the curve up to first cracking is similar to that of un-reinforced mortar. The descending part depends on the fibre reinforcing parameters, notably fibre shape, fibre amount and aspect ratio. The descending, or post-cracking, portion of the stress-strain curve shows the superiority in toughness of SFR-ECC over conventional un-reinforced cement-based composites. This is primarily because of the large frictional energy and fibre bending energy developed during fibre pullout on either side of a crack (fibre snubbing), and because of multiple cracks when they occur.

The test set-up for tension of cast composite specimens is shown in figure 17.

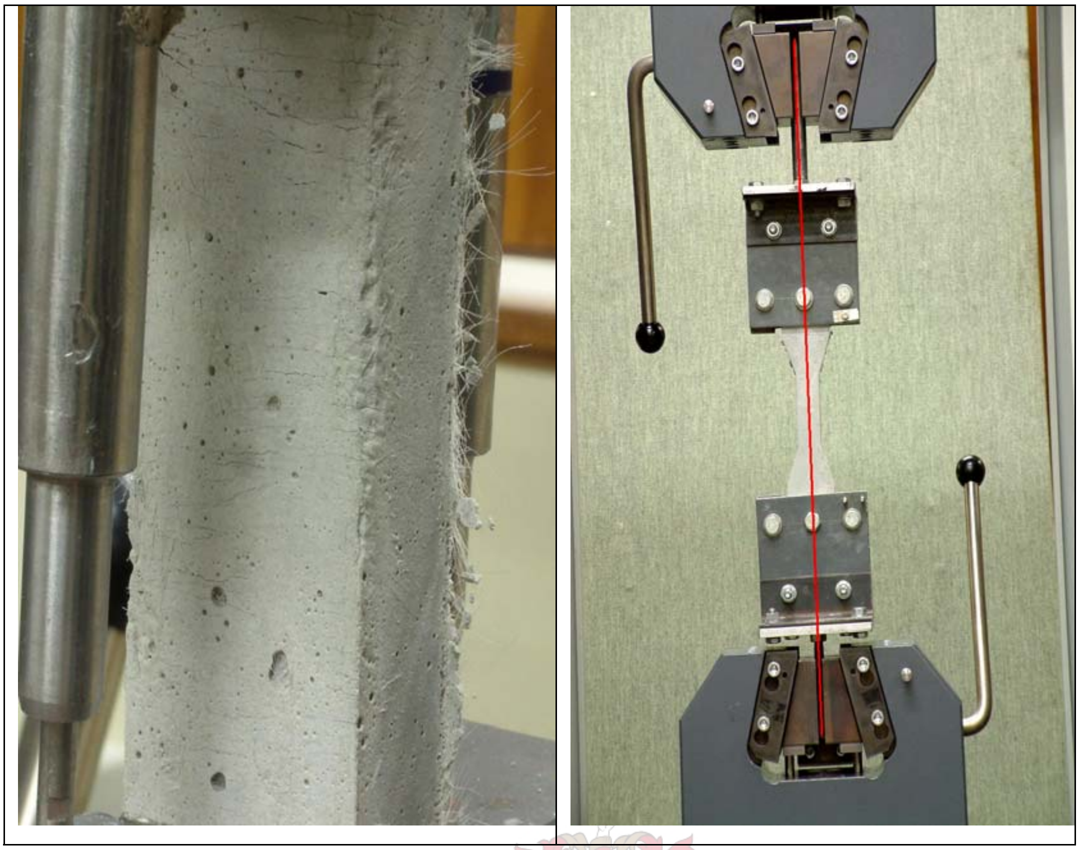


Figure 17: Zwick material testing machine tensile test setup

Tests were done on dog bone shaped specimens in a Zwick Z250 at a rate of 0,5mm/minute. Deformation was measured over an 80mm gauge length with an extensometer. The Spider8 data logger then converts the incoming data from the load cell and the extensometer deflection gauge to a data output file directly on a computer.

3.2.3 Flexural strength (indirect tension)

The influence of steel fibres on flexural strength of ECC is much greater than for direct tension and compression. Two flexural strength values are commonly reported. One value, termed the first-crack flexural strength, corresponds to the load at which the load-deformation curve departs from linearity. The other corresponds to the maximum load achieved, commonly called the ultimate flexural strength. From this maximum load, the so-called modulus of rupture is computed.

Three types of flexural failure modes have been observed in cement-based materials brittle, quasi-brittle, and ductile failure. Figure 18 provides a schematic illustration of these failure modes.

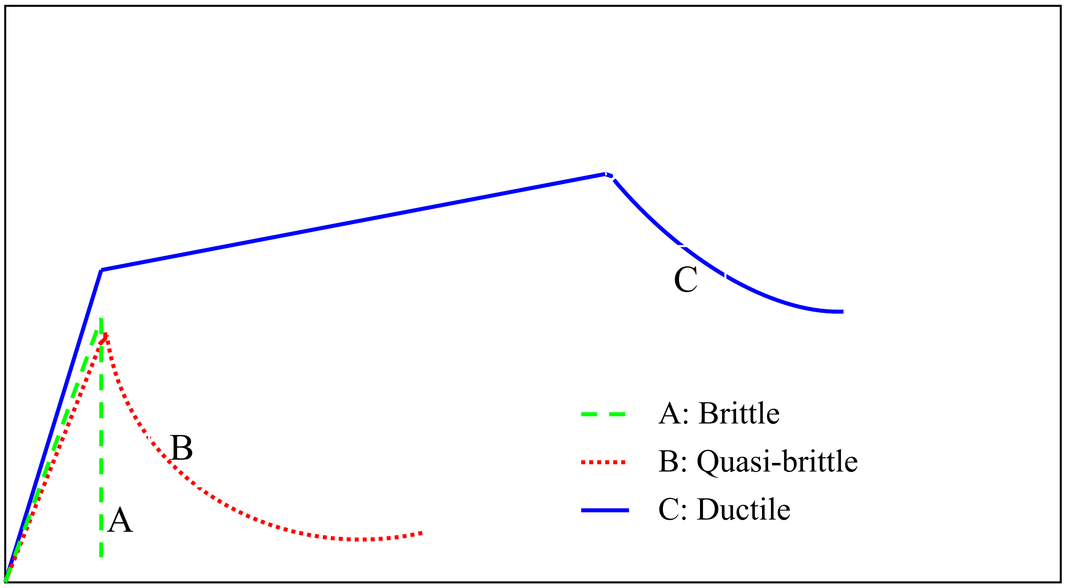


Figure 18: Three types of flexural response failure modes observed in cement-based materials

Normal concrete and cement pastes exhibit brittle tensile failure, while conventional FRC is characterised by quasi-brittle tensile failure. Brittle failure and quasi-brittle failures are both characterised by a linear stress-strain curve. Quasi-brittle material behaviour however has a softening tail after first cracking due to the bridging action of the fibres, and its toughness is enhanced due to the inelastic energy absorption in the post-peak region. Ductile failure is found in ECC and is characterised by its ability to sustain higher level of loading after first cracking while undergoing large deformation (curve C, figure 18).

4. Experimental Philosophy, Design and Procedure

In this chapter, the mechanical properties of ECC are discussed in terms of its response to uni-axial compressive and tensile loading, as well as flexural loading. Next, the particular demands of processing techniques like casting, extrusion and spinning on the ECC mix design are elaborated. Knowing these demands, the challenge of designing sufficiently equivalent mixes for these processes, to enable comparison of the process-induced mechanical properties is described in the section on the experimental philosophy. Thereby, the particular choice of ECC mix for each process is stated and clarified. Another factor in the commercialisation of ECC material products design is the cost. The material costs and viability analysis for the chosen mix design in this research are referred to in the appendix. Finally, the procedure of fabricating, curing and processing the specimens for the experiments are described.

This thesis research focuses on the orientation of the fibres and the influence of the manufacturing processes on the fibre orientation. Manipulation of rheological properties of ECC is required to modify it for the different manufacturing processes. In general, cast and vibration mixes must be highly workable, while mixes for spinning and extrusion should be dry and of high viscosity. Dry mixes for spinning and extrusion are distinguished by the aggregate and ingredient material particle fineness in general, with larger particles required for spinning to prevent stickiness, while this is less critical for extrusion mixes.

Further criteria should be kept in mind when designing ECC mixes. Through micro-mechanics based modelling, it has been shown that material and geometrical properties of the fibres, stiffness and strength properties of the matrix and the fibre-matrix interaction determine the multiple cracking, pseudo-hardening tensile response of ECC. These properties must be balanced, keeping in mind a particular application. In this way, particular ECC mechanical responses can be designed, like those shown in Figure 19. High uni-axial tensile strength can be obtained, at the price of lower ductility. Alternatively, uni-axial tensile strength can be sacrificed to gain large ductility, up to 5 %.

For both cases it is essential to avoid fibre breakage. Fibre breakage is known to produce more brittle tensile behaviour than fibre pull-out from ECC matrices (Shah et al., 1997 & 2003; Li et al., 2001). This is illustrated in Figure 20 by two specimens tested in another project (Song and Van Zijl, 2004) of the same research programme of which this thesis forms a part. The figure shows direct tensile test results of similar specimens, containing PVA fibres of length 13 mm and diameter 0.020 mm. However, for one specimen, ECC 2 in Figure 20, 70% of the cement was replaced by fly-ash, in effect lowering the tensile strength. Fibre pull-out accompanied this weaker, but tough response, while fibre breakage was audible in the case of the stronger matrix, ECC1 in Figure 20. It is clear that the latter, stronger matrix has lower deformability. Although both specimens were produced by standard casting and vibrating, this example illustrates that brittle failure is associated with stronger matrices. Fibre breakage leads to such brittle failure. As an example, SFRCC behaviour is compared to ECC 1 and ECC 2 (Figure 20). In the case of extruded ECC, products of lower porosity are produced, associated with denser, stronger matrices. Thereby, the same effect as shown in Figure 19 may arise, by comparing the tensile mechanical responses of identical matrices, but from different fabrication processes of casting (weak, tough) and extrusion (strong, brittle).

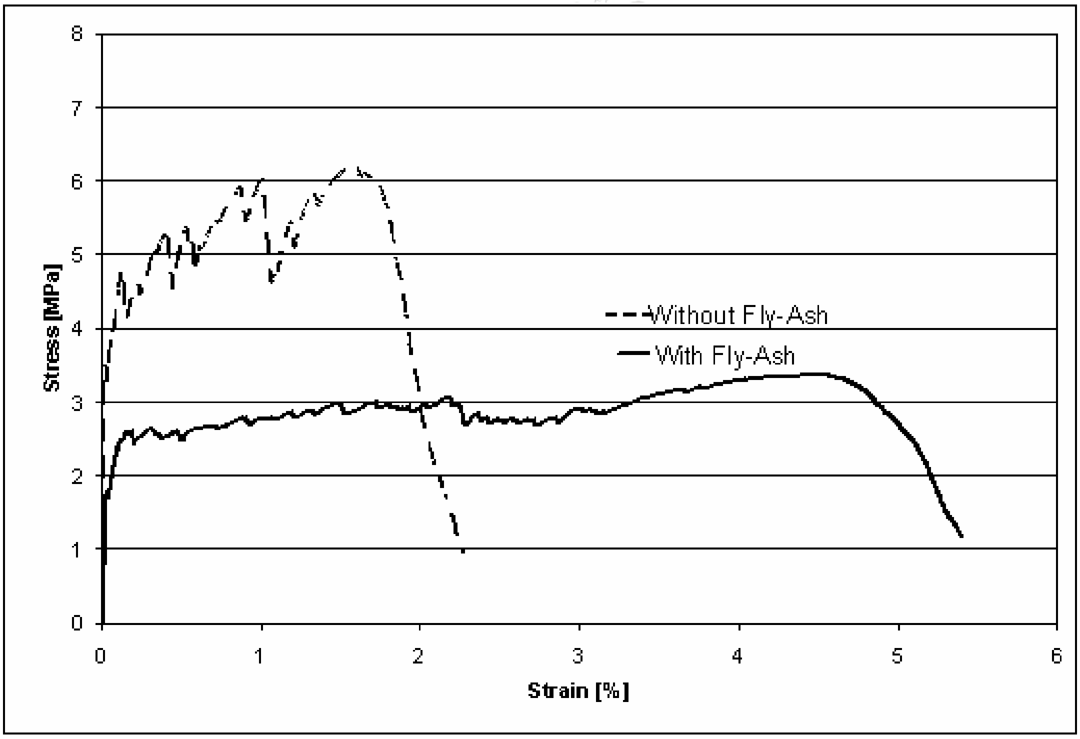


Figure 19: Fibre failure modes: strong, brittle matrix (without fly-ash) and weaker, ductile mix (with fly ash)

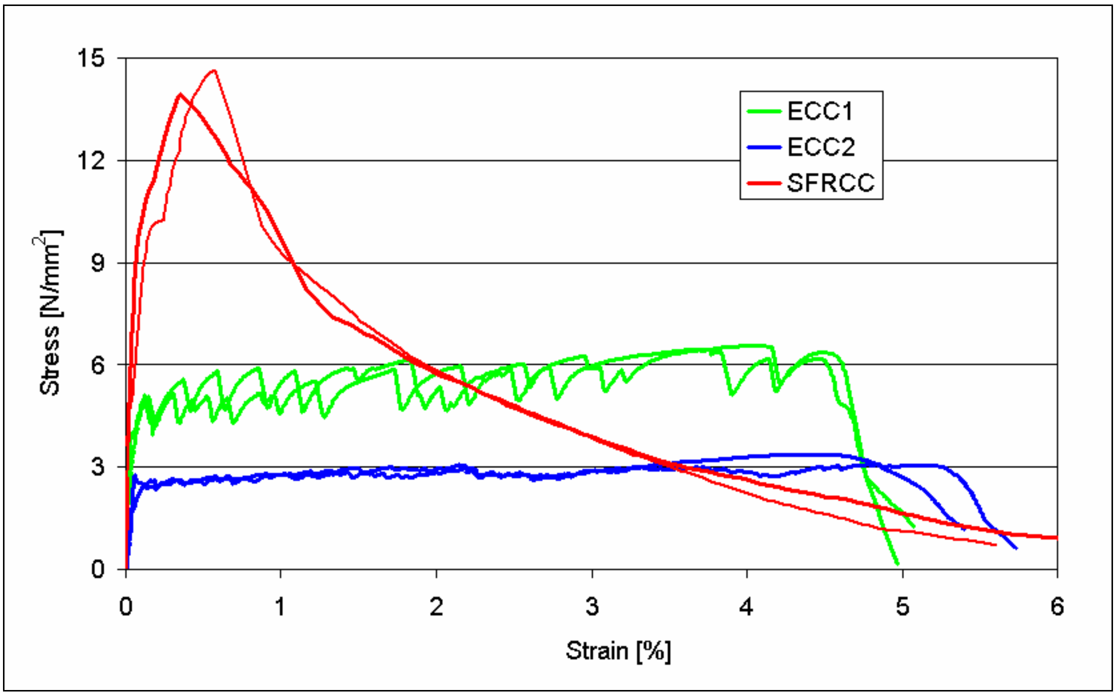


Figure 20: Various ECC materials types' direct tensile response

However, the distinction between strength and toughness is not simple. Material with high direct tensile strength may lead to equal or lower structural resistance than material with weak, but ductile tensile behaviour when elements of such materials are subjected to high stress gradients. This is illustrated in Figure 20 - a simplified analysis of the performance of the specimens reflected in Figure 20, but under more general loading, in this case three point bending. This loading produces stress gradients and reveals that the tough uni-axial tensile response may cause a modulus of rupture (MOR) of several times the composite tensile strength. However, this cannot be achieved by the less ductile matrix, as tensile softening degradation upon higher straining of fibres far from the neutral axis, limits the ultimate resistance in flexure.

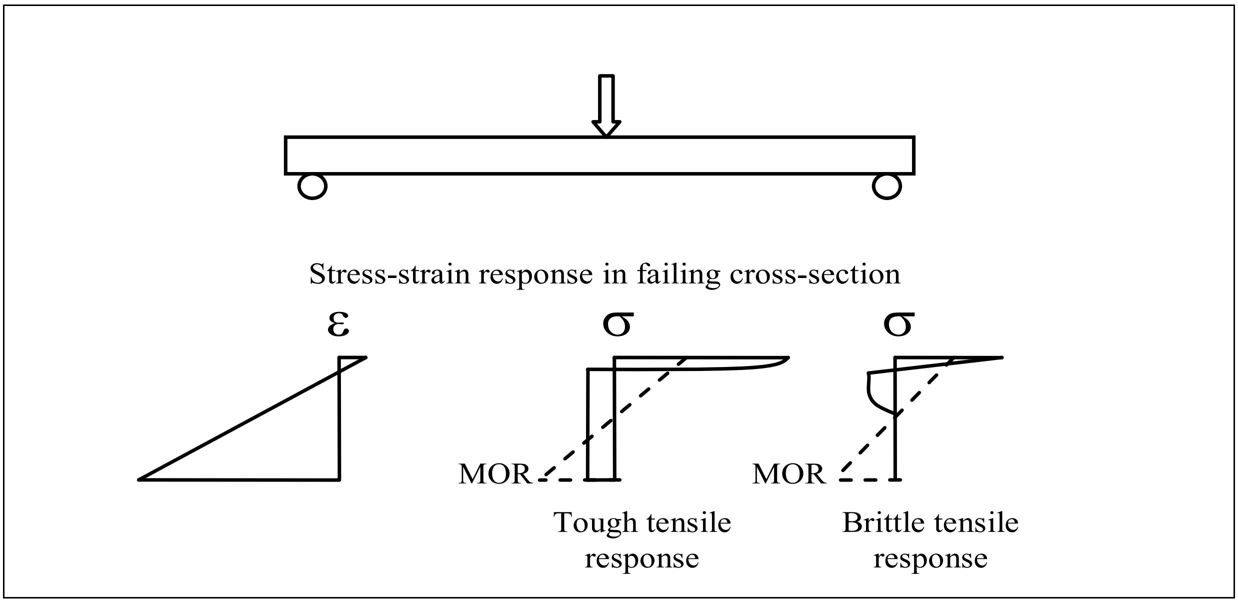


Figure 21: Tensile strain-limited flexural resistance of brittle material

The flexural resistances of the two materials of Figure 20 are computed as shown in Figure 21. For the two matrices of the following relations are approximately true: $\sigma_{cu} / \sigma_{tu} = 6.5$, E = 350.$\sigma_{cu}$, where σ_{cu} is the composite compressive strength, σ_{tu} the composite ultimate tensile strength and E the composite Young's modulus. Sectional equilibrium and ultimate moment calculation with the simplified stress distribution for tough tensile response in Figure 21 reveal that an ultimate tensile strain $\varepsilon_F = 2.9\%$ is required to reach the full potential in bending, i.e. to realize stress transfer in all fibres in the cross-section at ultimate resistance. This is achieved by the FA specimen (Figure 21) but not by the stronger matrix without FA. The calculation predicts a ratio of MOR / σ_{tu} = 4.5. Experimental results reported by Peled and Shah (2003) show this ratio to be 3.5 for extrudates containing FA, but only 2.0 for stronger extrudates, which contain no FA. As their results were obtained by the comparison of bending results only, i.e. by comparing fibre-reinforced composite flexural response to that of matrices without fibre reinforcement, the uni-axial tensile response is not known, which prevents complete validation of the above analytical prediction. Nevertheless, the reduction in the bending resistance to axial resistance ratio shown by their results goes a long way to prove that this trend accompanies increased brittleness.

In the light of the above-mentioned influences on ECC material properties, an experimental program was designed and executed to, on the one hand develop mixes suitable for particular processes and, on the other hand, develop processes suitable for processing the material. The latter was necessary because, despite the level of maturity of extrusion processes for manufacturing of building products from other types of material, for instance clay or plastics, the application to ECC is currently in the developing stages. It is being actively studied by several research groups, for instance Shah et al., 1997, 1998, 2003; Stang et al., 1999; Kearsley et al., 2004; Li et al., 2003.

The rest of this research studies the mechanisms which influence, or even govern, the tensile response of ECC, with particular reference to the influence of extrusion on fibre orientation and fibre-matrix interfacial zone densification.

4.1 Mix design

Our aim was to optimise the mix design to achieve a strong and/ or ductile matrix for a specified purpose and for a specified manufacturing technique. Furthermore, the goal of the research project was to study the processing influence on the mechanical response. Therefore, a basic mix was the design task, suitable for all production processes. In this section it is described how and to which degree of success this was undertaken.

It was intended to be able to distinguish the mechanical enhancement by fibre orientation and matrix densification, both of which are brought about or significantly dependent on the particular processing method. So, it was decided to maintain a basic matrix for all processes. This mix maintained the constituent mix proportions as tabulated below.

Water:binder ratio	0,25
aggregate:binder ratio	0,5
fly-ash/binder ratio	0,5
silica fume/binder ratio	0,05
volume fibre	0,03

Table 5: Basic constituents mix proportions for casting and piston extrusion

Furthermore, steel fibre was used in nearly all cases, to enable visualisation of fibre dispersion and orientation by affordable methods, like X-Ray methods. Parallel studies in the group at the University of Stellenbosch focussed on PVA fibres. Also in this study some mixes contained PVA fibres, namely spinning and piston rheology tests conducted at the University of Pretoria.

4.1.1 Mix design for extrusion process

Quality extrusion requires a balance between ingredient materials, and the processing and properties thereof. In the case of extrusion, extrude-ability and shape stability put contradicting demands on mix design. The material must be rigid enough to retain the shape fixed by the die geometry, yet must be soft enough to flow into and through the die of the extruder. With the addition of additives in the form of methyl cellulose and plasticizers that impart a high viscosity, yet good dispersion of ingredients, a dough-like mix results, which ensures both extrudability and retention of the required shape.

Figure 22: Auger extrusion of a pipe

Figure 23: Piston extrusion of a pipe

4.1.1.1 Rheological characterization and extruder design considerations

Extruded ECC (EECC) can be efficiently developed with the aid of an extrusion rheometer (Shah et al., 1999). Since the ECC pastes are difficult to characterize with normal concrete rheology models, a more appropriate technique was adopted to characterise extrusion mix designs. A small-scale piston extruder, sensitive to compositional changes, was built and utilised as a rheometer in characterizing ECC.

The rheometer, or small scale piston extruder, enables measurement of extrusion pressure and visualisation of segregation and extrudate (dis)continuity, leading to expression and understanding of the rheological properties of the mix. This all can conveniently be done with small quantities of mix samples, required especially for new ECC mixes before full-scale production.

The mini piston extruder was constructed in a cage-frame to have the main load-carrying steel members in tension whilst eliminating eccentric loading. The plunger closely fits inside a 50mm barrel section (hollow round tubing) with adjustable centred die connections. The entire piston arrangement was thus supported by means of a 'portal-like' frame constructed of mild steel, allowing easy assembling and future alterations of the whole arrangement.

Figure 24: The mini round-bar extruder

Figure 25: Mini round-bar extrusion

4.1.1.2 Mini piston extrusion tests on the mini piston extruder to develop an extrude-able mix

STEP1: The aim was to obtain the optimum amount of viscous agent (VA) for the mix. Workability for the whole range of tests was maintained by adjusting the water: binder ratio, in other words adding more water when increasing the amount of VA. The VA was adjusted while the Super-plasticizer (SP) was kept at a constant 1%.

Table 6 lists the respective VA percentages:

VA (% of binder mass)	SP (% of binder mass)	Observed
0	1,0	Not extrude-able; not included in measured data
0,3	1,0	The mix exhibits bleeding when extruded, and extrusion forces necessary to extrude the fibre reinforced extrusion mix are very high. When the mix starts bleeding, a Force vs. Deflection asymptote emerges.
0,6	1,0	Signs of bleeding behaviour dissipates
1,0	1,0	No bleeding and the mix is easy to extrude
2,0	1,0	No bleeding, easy to extrude, and the mix becomes costly

Table 6: Influence of VA on extrusion, as observed with the piston rheometer

The force-displacements measurements for piston extrusion tests are shown in Figure 26.

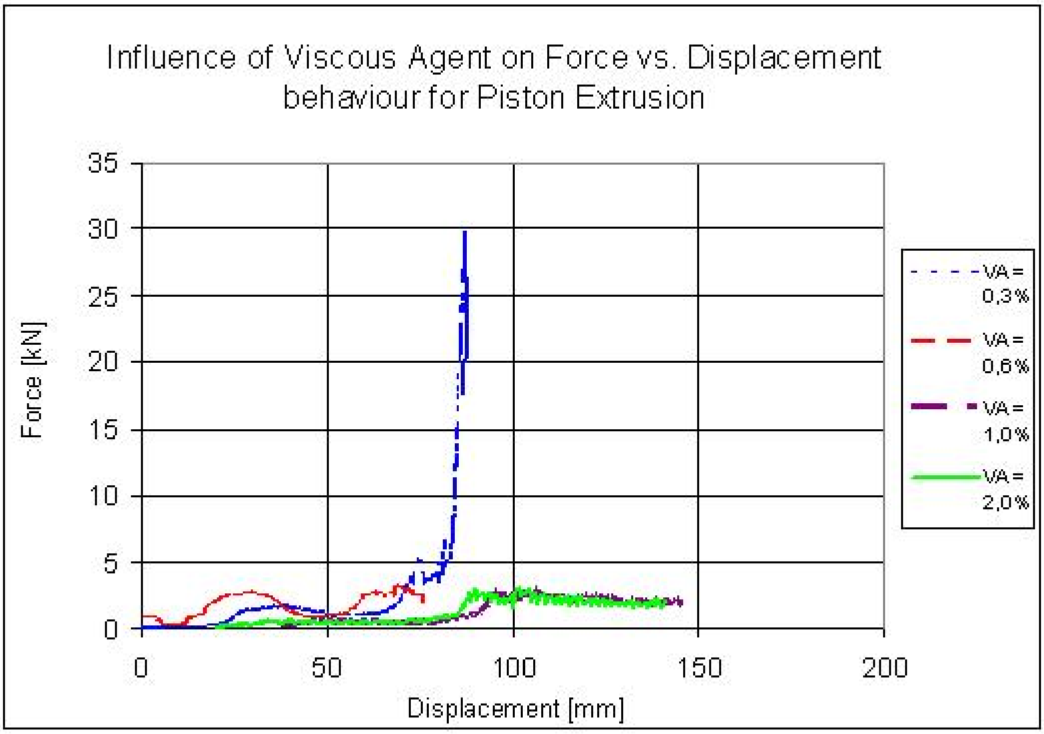


Figure 26: Influence of VA on the force needed to extrude a 20 mm diameter cylindrical specimen

Successful extrusion was found in terms of non-segregation and extrusion force required for the range of 1% to 2% viscous agent. From a cost point of view, it made sense to opt for the 1% VA. It is not only less expensive, but also reduces any other unknown short or long term effects, which this chemical admixture may have on ECC. One potential danger is retardation, which was an observed effect of combinations of high VA and SP.

The important finding is that, although the viscous agent assists in dispersing the fibres and in doing so, preventing fibre clumping and indirectly reducing extrusion force, it also aids in retaining the water in the extrusion mixture whilst under pressure.
The optimal water: binder ratio was established to be 0,25. This was decided upon in STEP 1 for the viscous agent of 1% and the super-plasticizer of 1%.

STEP2:
Finally the role of super-plasticizer was studied systematically. Table 7 contains the summarised comments on the range of SP amounts used in the mix.

VA (% of binder mass)	SP (% of binder mass)	Observation
1,0	0,5	the mix was a little on the dry side for extrusion, not workable enough
1,0	1,0	the mix exhibit excellent extrude-ability and shape retaining
1,0	1,5	very extrude-able, but not capable of shape retaining
1,0	2,0	once again, easy to extrude but not shape retaining

Table 7: Influence of SP on extrusion observed with the piston rheometer

The water:binder ratio was adjusted (increased) for the various chemical additives compositions to maintain (visually) the same workability.

The super-plasticizer of 1,0%, the viscous agent of 1,0% and the water:binder ratio of 0,25 was established to optimise the workability of piston extrusion (kindly refer STEP1).

This was how the mix was developed for the piston extrusion (and extrusion in general) process. The final mix, in agreement with Table 5, but with inclusion of specific amounts of additives for extrusion, is summarised in Table 8.

This mix satisfies the minimum water:cement ratio criteria of 0.37 for 100% hydration of the cement, because the water:cement ratio is 0,56.

Constituent	Amount	Density [kg/m^3]	Amount - Mass/ Volume [kg/m^3]
Water	Water: binder ratio = 0.25	1	310.1
Binder:			
Cement	Cement:binder ratio = 0.45	3.14	558.1
Fly Ash	Fly ash:binder ratio = 0.50	2.8	620.1
Silica Fume	Silica fume:binder ratio = 0.05	2.9	62.0
Aggregate – Philippe Sand	Aggregate:binder ratio = 0.50	2.7	620.1
Super-plasticizer (SP)	SP: binder ratio = 0.01	1.2	12.4
Viscous Agent (VA)	VA: binder ratio = 0.01	2.0	12.4
Fibres – Steel Fibres	Steel fibre:mix volume ratio =0.03	7.85	235.5
OR			
- PVA Fibres	PVA fibre:mix volume ratio =0.03	1.3	38.8

Table 8: Final mix design sheet for the extrusion ECC mix

4.1.2 Mix design for castings process

Casting requires the mix to be workable and flowable especially for application in moulds for thin-walled specimens like pipes. It must be flowable and compactable, without leading to segregation, in order to get continuity of the cast composite in the moulds.

The addition of fibres also tends to increase the viscosity of the cement-based matrix, which makes it very difficult to handle and place. For this reason, in bulk construction and with conventional mixing techniques, it is only possible to use short fibres (shorter than 25mm) in volume fractions of not more than 3 percent.

The aim was to get the extrusion mix castable in order to eventually produce plate and pipe cast specimens, and to compare results in three point bending with that of extruded plate and pipe specimens.

Figure 27: Cast processing of a pipe

The cast mix design requires a much more workable ECC mix design than the extrusion mix. Some tests were conducted to ascertain the water, super-plasticizer and viscous agent content needed to achieve the flowable cast mix. It was desirable to have the same water/binder ratio for both extrusion and casting. With the aid of the cast apparatus shown in Figure 27, some experimentation was performed and is briefly described here.

a) By keeping the VA and the SP the same as for extrusion, namely 1% each, the water:binder ratio had to be adjusted from 0,25 to 0,37 to get it sufficiently workable. Since any change in the water:binder ratio influences the strength characteristics of the composite, this change was too drastic and consequently the mix was not used.

b) With the second series of tests, the water:binder ratio was kept at 0,25, while the SP and the VA were varied individually in steps of 0.1% (i.e. 0.0%, 0.1%, 0.2%....) in order to see how much VA and SP would still leave the mix cast-able. The outcome was that 0,1% VA and 2,5% SP were required. This is considered as a limit value of VA to still providing fibre dispersion and restraint to segregation. The SP is on the other extreme end of the range that should be used. Nevertheless, these values were accepted in the light of the goal of keeping the rest of the matrix the same as for extrusion. The final mix details for casting are tabulated in Table 9 below.

Constituent	Amount	Density [kg/m^3]	Amount - Mass/ Volume [kg/m^3]
Water	Water: binder ratio = 0.25	1	310.1
Binder:			
Cement	Cement:binder ratio = 0.45	3.14	558.1
Fly Ash	Fly ash:binder ratio = 0.50	2.8	620.1
Silica Fume	Silica fume:binder ratio = 0.05	2.9	62.0
Aggregate – Philippe Sand	Aggregate:binder ratio = 0.50	2.7	620.1
Super-plasticizer (SP)	SP: binder ratio = 0.025	1.2	12.4
Viscous Agent (VA)	VA: binder ratio = 0.001	2.0	12.4
Fibres – Steel Fibres	Steel fibre:mix volume ratio =0.03	7.85	235.5
OR			
- PVA Fibres	PVA fibre:mix volume ratio =0.03	1.3	38.8

Table 9: Final mix design sheet for the cast ECC mix

4.1.3 Mix design for spinning process

For the spinning process an attempt was made to keep a common mix design as far as possible to be able to compare products of the different processes. However, the need for larger aggregate size for a practical spinning was reconfirmed. The practical minimum upper limit of the aggregate is more than 6mm. As stated in section 3.1.1, such large aggregate particles prevent good matrix-fibre interaction required for strain hardening of ECC materials. This leads to a decrease in the ductility, whether it is strain hardening or strain softening.

Figure 28: Spinning processing for pipes

For this reason we only looked at the influence of the spinning process on fibre orientation of the composite. We did not investigate adjusting of the mix for the spinning process. Figure 28 illustrates the spinning process.

4.2 Experimental procedure

Here follows complete detail of the manufacturing processes. The table below gives an outline of the various procedures conducted (relevant to this research programme).

	Description of procedure:	**Applicable process:**
4.2.1	Mixing of FRC/ ECC	All processes
4.2.2	Specimen types	All processes
4.2.3	Specimen manufacturing	All processes
4.2.4	Stripping of moulds	Cast tensile moulds (dog-bones), cast three point bending beams, cast pipes, cubes for compression tests.
4.2.5	Curing procedures	All processes
4.2.6	Drying	All processes
4.2.7	Preparation for testing	All processes

Table 10: Outline of experimental procedure

4.2.1 Mixing of ECC

Mixing procedure that was used is as follows:

- Before starting with the mix, 4 tensile moulds and 3x 100mm cubes were oiled.
- Quality control was very important, and we made sure of the correct weight for each substance.
- The sequence by which substances were added was of great importance, as was the mixing times. The following diagram was used as a guide:

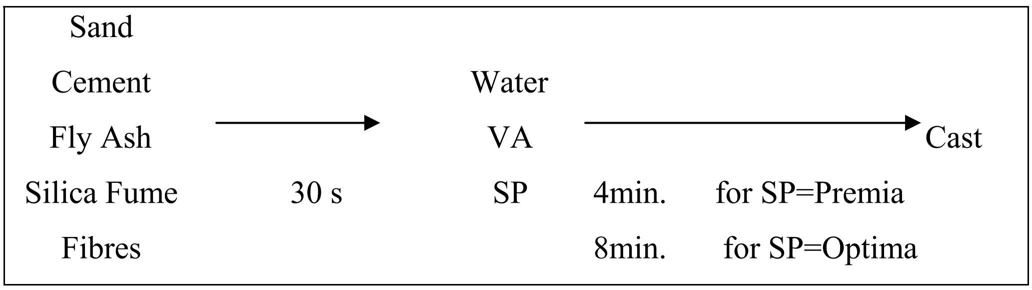


Figure 29: Mixing procedure diagram

- First all dry ingredients, including aggregate, binders and fibres were added and mixed for 30s
- At the same time, the VA and SP were mixed with the water before adding it into the mixer
- Then, the water + SP + VA mix was added
- The mixing times were 4 minutes in the case of using Chryso Premia 100 super-plasticizer, while 8 minutes mixing time is required in the case of using Chryso Optima 200 super-plasticizer

  In this research, only Premia 100 super-plasticizer was used.
- When the mixing was finished, the fibre dispersement was quantified using the following as a guide:

  1. Very clumpy, i.e. fibres tend to clump together; fibres are not evenly dispersed and one can feel the fibres between your fingers when using your hands to feel the texture of the mix
  2.
  3. Feel a few fibres in the mix
  4.
  5. Very smooth – no fibres can be felt when one uses your hands to feel the texture of the mix

  We physically used our hands to feel the texture of the mix.

4.2.2 Test specimen types:

The specimen types that were manufactured for the experimental procedure are as follows:

- Cast composites - cubes (100mm x 100mm x 100mm)
- Cast composites - dog-bones (30mm x 15mm nominal narrow section 80mm long, before tapering out for grip)
- Cast composites - plates (70x15x500mm)
- Cast composites - pipes (150mm nominal diameter and 15mm wall thickness and 700 mm long)

- Piston extruded plates - (cross section 70mm x 15mm)
- Piston extruded pipes - (150mm nominal diameter and 15mm wall thickness, length at least 700 mm)
- Auger extruded plates - (80x15mm)
- Auger extruded pipes - (85mm nominal diameter and 15mm wall thickness)
- Spinning pipes - (175mm diameter and 20mm wall thickness)

4.2.3 Specimen manufacturing:

Different processes called for unique test specimen manufacturing techniques, and through trial and error some manufacturing criteria to assist and support the process manufacturing were established. Some guidelines to execute the process in a consistent manner were also set.

4.2.3.1 Cast composites – cubes, dog-bones, plates, pipes

All the moulds were oiled before commencement of casting.

Careful placement of the mix in the tensile moulds (dog-bones) was always important as this has a direct effect on the results of the tests: it was carefully placed in the moulds starting from the middle outwards.

Vibration frequency of all the moulds for a specific cast composite type was consistent and the only real criterion was a smooth cast finish.

Vibration of the dog-bones:

- Vibration frequency provided a smooth finish.
- Vibrated until the concrete settled.
- The top covering steel plate was carefully placed onto the mould.
- Afterward, we vibrated again.

Vibration of the cubes, plates:

- Vibration frequency provided a smooth finish.
- Vibrated until the concrete settled.
- This provided a smooth level finish at the top of the mould.

Vibration of the pipe moulds:

- Vibration frequency was at 100% of the vibration table amplitude.

- The base-plate of the mould apparatus was clamped to the vibrating table.
- Vibrated while the mix was added into the top of the mould.

After we finished casting, the moulds were immediately placed in the climate control room, where the temperature was controlled at 23°C.

4.2.3.2 Piston extrusion

The piston extrusion manufacturing process is a discontinuous process and requires multiple (back-to-back) mixing when a large volume is extruded. This was necessitated by the dough-like mix that dries out quickly and hence forms “cold” joints at reload points, i.e. weak joints in the matrix due to a discontinuity in the manufacturing process.
For example, we split the required volume up in two identical batches when extruding three pipe specimens of 1m length each, 150mm ID and 180mm OD. For smaller mix volumes of less than 20 litres (for example extrusion with the plate piston extruder) one mixing batch was sufficient.
The mix volume required was calculated from the waste material that remained in the transition and die of the extruder together with volume of the specimens to be manufactured. The material that accumulated in the transition and die of the extruder formed part of the extrudate following the first extrusion cycle, but remained in the apparatus after the last extrusion cycle. This material was wasted and removed in the cleaning process.
The moulds for the pipe extrudate and the platform for the plate extrudate were oiled before commencement of tests to provide a smooth surface to catch the extrudate on, and it also assists in providing a non-sticky surface when the extrudate is hardened.

4.2.3.3 Auger extrusion

Unlike piston extrusion, auger extrusion is a continuous process. Since the same dough-like mix is used in auger extrusion, it is recommended that the mix is also divided in smaller batches to avoid drying of the material.
It was ensured that the material was fed into the pug mill (or opening) continuously to avoid discontinuities in the extrudate. The moulds for the pipe extrudate and the platform for the plate extrudate were oiled before extrusion.

4.2.3.4 Spinning

The spinning process requires depositing the material from the sides (either by hand or with a spade) until the centrifugal forces levelled out the material and it becomes flush with the edges. The edges of the mould determine the wall thickness of the spinned pipe. The material is mixed in one batch.

4.2.4 Stripping of the moulds

- Cubes were stripped one day after casting.
- Tensile and beam moulds, as well as the mould for the cast pipe, were stripped 2 days after casting.
- Cube, tensile and beam moulds, as well as the mould for the cast pipe were cleaned, oiled and reassembled after the composites were taken out.
- Tensile moulds (dog-bones) were stripped in the following manner:

1. Pull out of the two studs.
↓
2. Carefully remove the top.
↓
3. Loosen the bottom by removing the top bolts, while keeping the sides fastened.
↓
4. Slide the specimen on the bottom to loosen it from the bottom plate.
↓
5. Remove the sides carefully by removing the horizontal bolts on the side.
↓
6. Carefully pick up the specimen using two hands.
↓
7. Clean, oil and reassemble the mould (before stripping the next mould)

Figure 30: Tensile moulds stripping procedure

4.2.5 Curing

Curing of the specimens plays a very important role in the hydration process and influences the structural strength and durability of the composite. Consistency of the curing process is important.

After stripping the moulds of a mix, the tensile, cubes and beam specimens were marked and immediately placed in water in the room where test specimens were cured immersed in water.

- The cubes, tensile and beam specimens of a specific mix were kept grouped together in the water. In this room the moulds were marked with pieces of paper.
- The water level of the container where the cube, tensile and beam specimens were kept, was checked daily to make sure that specimens were always submerged.
- In the case of the cast pipe specimens, the pipes were placed in a climate control room where the relative humidity was kept at 100%. It could also be cured in a steam curing room.
- The cube specimens were immersed in the water for 21 days.
- The tensile and beam (three point bending) specimens for the cast, auger and piston extrusion processes were immersed in the water for 28 days.
- The pipe specimens, cast and extruded, were kept in the water for 21 days.

4.2.6 Drying

The cubes, tensile and beam specimens were taken out of the water and the cast pipe specimen taken out of the moisture room 2 hours before testing.

4.2.7 Preparation for testing

Here follows an elaboration on the description of the preparation that went into the various testing methods for the different specimens as listed in paragraph earlier.

4.2.7.1 Tensile testing (direct tensile tests): cast composites - dog-bones

The direct tensile tests were done after the 28 days curing period. The setup of the LVDT (linear variable displacement transducer) displacement measurement apparatus in the earlier stages of the research and the extensometer strain measurement apparatus towards the end of the research is described below.

LVDT measurements

Gluing the LVDT clamps for tensile specimens preparation before testing, where LVDT measuring is one way to measure the strain of the tensile specimen.

The procedure of preparing the tensile specimens for the direct tensile tests if one decides to use LVDT measurements is:

- Two days before testing clamps are glued to one side of the tensile specimen.
- One day before testing clamps are glued to the other side of the tensile specimen.
- It is import to make sure that clamps are glued square, otherwise test results will not be accurate.
- The clamps can be glued at an earlier stage, but not later than the specified time.
- The placement of the clamps is shown in figure 31.

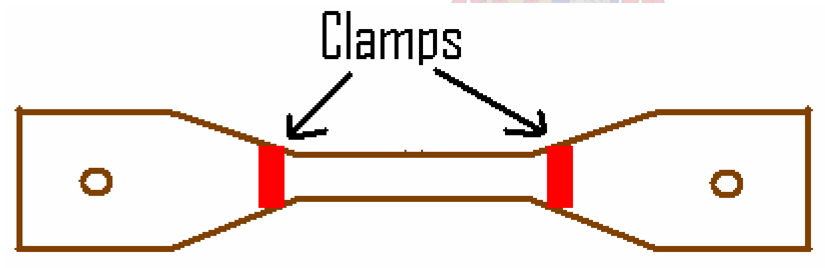


Figure 31: LVDT tensile test clamps positioning

Extensometer measurements

An alternative device for measuring the deformation is an extensometer. This was used in the latter part of the research and applies to all the measurements reported in chapter 5. The advantage of using an extensometer is that no gluing is required for the LVDT.

The tensile test is very sensitive, thus extreme care was taken while testing the specimens.

For both the LVDT and the extensometer measurement techniques, the following procedures were followed during testing:

- Before any testing commenced, the LVDT's or extensometer were calibrated (which ever method was our choice of strain measurement).
- Before specimens were tested, the following dimensions were measured to the nearest 0.5 mm:
    1. The thickness at the middle.
    2. The width at the middle.
- The following procedures were followed when direct tensile tests were done:
    1. Dimensional measurements were taken as described earlier.
    2. The tensile frame bolts were fastened slightly (with hand) with the bottom side of the specimen towards the person testing.
    3. The bottom Zwick clamp were closed to let the specimen hang straight, making sure that the bolts of the clamps are not resting on the Zwick clamps.
    4. The Zwick clamps were closed slowly and carefully, the bolts of the bottom clamp were fastened and then slowly fastened the top bolts, while making sure there was no load applied on the specimen by looking at the reading on the Zwick computer. If load increased, the load was released by opening and closing the bottom Zwick clamp.
    5. The LVDT's or the extensometer was fastened carefully and then the bottom clamp of the Zwick was released.
    6. The force measured by the built in load cell was set to zero. The readings are interpreted by the Zwick computer. The Zwick load cell is accurate to the nearest newton reading.
    7. The bottom Zwick clamp was then closed.
    8. The measurement on the Spider computer started.
    9. The force reading on the Zwick computer and the displacement on the Spider computer was close to zero.
       If not – the procedure from step 6 was repeated.
    10. Testing of the Zwick computer controlled procedure started.
    11. Digital photographs were taken periodically of the specimens.
    12. If the load reading decreased to a load less than half the peak load, the measurements on the Spider computer were stopped.

13. The data was then exported and the first 4 channels were selected. The output data format used was ASCII (with header).
14. The Zwick computer was then stopped.
15. The LVDT's or extensometer was then removed, and the specimen removed from the clamps afterwards.

4.2.7.2 Compression testing: cast composites - cubes

- The compression tests were done at an age of 21 days.
- Compression tests were performed according to SABS 863:1994.

4.2.7.3 Flexural tests (three point bending test): cast composites – plates, piston extruded plates and auger extruded plates

Three-point flexural tests were carried out to characterise the flexural response of extruded and cast composites (refer figure 32).

Figure 32: Three point bending setup for flexural tests

The length of the span between the two supports was 400mm. The specimens were loaded at the centre and had cross sections of 70mm wide by 15mm thick. Most of the samples were tested at 28 days, while a few were tested at 7 days. Three-point bending tests were conducted by displacement control at the rate of 3mm/minute. The Spider8 data logger then converted the incoming data from the Zwick load cell and the LVDT deflection measurement device at mid-span, to a data output file on the computer.

The flexural bending tests were performed at an age of 28 days.

4.2.7.4 Pipe bending tests: cast composites – pipes, piston extruded pipes and auger extruded pipes

- All the pipe specimens listed above were tested according to the SABS 819-2003 rev.1 standard: “Fibre-cement pipes, couplings and fittings for sewerage, drainage and low-pressure irrigation”.
- Flexural tests were performed according to paragraph 5.6 in the SABS 819-2003 standard, and were conducted after 21 days’ curing period.
- Special reference is made to the 4th year thesis document of Mr C. Jordaan (2003), thesis number S17 for detailed apparatus measurements and setup.

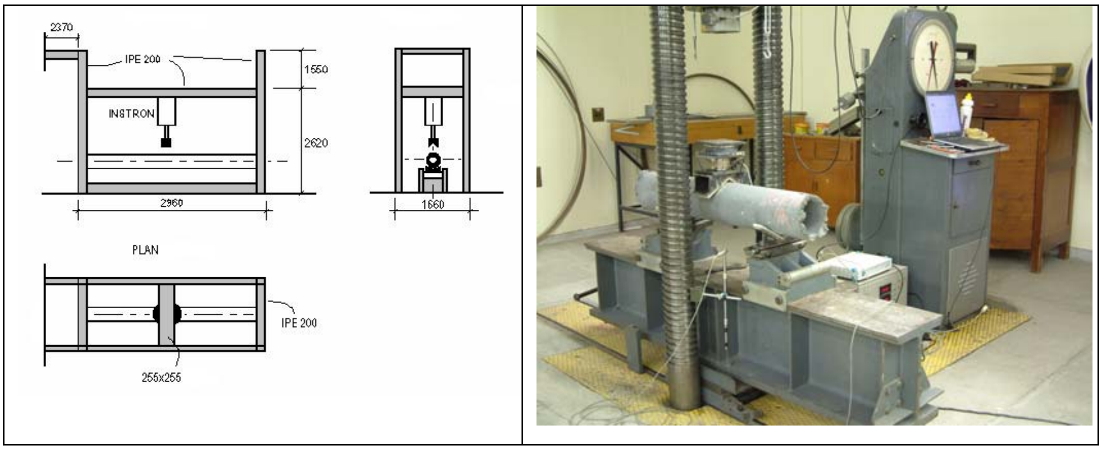


Figure 33: Three point bending test setup for fibre reinforced pipes

4.2.7.5 Pipe crushing tests: cast composites – pipes, piston extruded pipes and auger extruded pipes

- All the pipe specimens listed above were tested according to SABS 819-2003 rev.1: "Fibre-cement pipes, couplings and fittings for sewerage, drainage and low-pressure irrigation".
- Flexural tests were performed according to paragraph 5.4 in SABS 819-2003 and were performed after 21 days' curing period.
- Special reference is made to the 4th year thesis document of Mr C. Jordaan (2003), thesis number S17 for detailed apparatus measurements and setup.

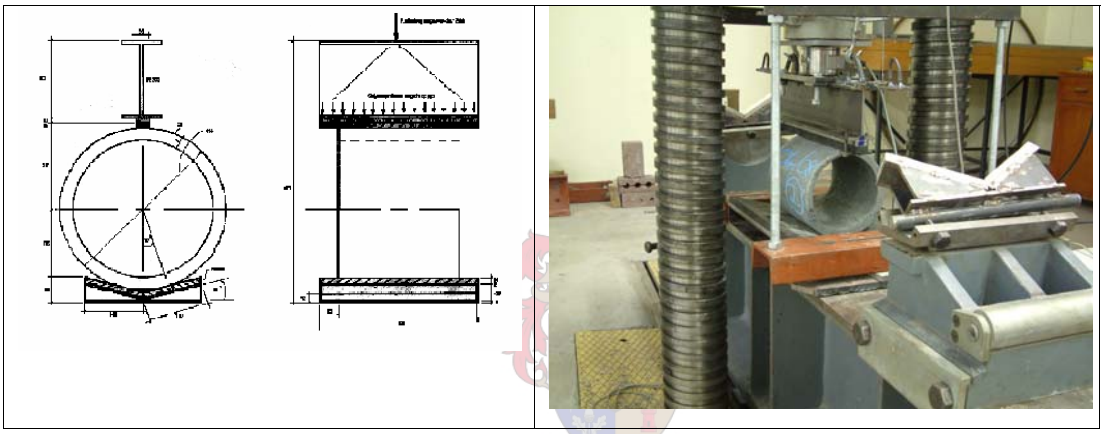


Figure 34: Crushing test setup for fibre reinforced pipes

4.2.7.6 Density measurement: all manufactured specimens have reference.

- Density tests were performed on all the manufactured plate and pipe specimens after the mechanical tests in 4.2.7.1, 4.2.7.3, 4.2.7.4 and 4.2.7.5 were performed.
- Density specimens, 50mm width, and 70-110mm length, were cut out of the tested specimens.
- The density specimens were then put in a drying oven at 60°C for 24 hours. This temperature and duration was found experimentally to ensure no more wet reduction due to moisture loss or drying.
- The weight of the dried specimen was measured.
- The tests were performed with a vase with cone-shaped lid to eliminate the meniscus error.

- Separately, the vase was filled with water and the weight measured, call it 'x'.
- The specimens were then taken out of the oven and the dry weight (W1) measured.
- Then, the specimens were submerged in the water in the vase, and the vase was filled to the top. This total weight was measured (W2).
- Displaced by the submerged specimen, the volume of the specimen could easily be obtained with the following formula:

$$\frac{(x + W_1) - W_2}{1000} = Volume_{specimen} \quad (7)$$

- After the volume and the dry weight of the specimens were determined, the densities were calculated:

$$Density = \frac{Weight}{Volume} \quad (8)$$

5. Development of Piston Extrusion Process

The purpose of this chapter is to describe the results of basic research conducted on piston extrusion, also known as ram extrusion. The goal of the research was to successfully develop and employ this method to fabricate products of ECC material. The final selection of extruder geometry is also discussed in this chapter.

Piston extrusion is a manufacturing process whereby material is compacted into a solid cylinder and then pushed out through a die orifice in the front of a shaped die. The process of extrusion converts a suitable raw material into a product of specific cross-sectional shape by forcing the material through a die or orifice under controlled conditions.

5.1 Design concepts, parameters and considerations

At the onset of determining the design parameters of the piston extruder, the piston rheometer (refer 4.1.1 and Figures 24 & 25) was built using design parameters found in the literature about similar rheological studies of ceramic and other materials. Special reference was also made to the research paper on the use of extrusion rheometry of Shah et al. (1999).

It is important to bear in mind that some parameters play a significant role in the design of a piston extruder, and that it is essential to understand how these parameters interact in order to calculate the necessary capacity of the extruder, i.e. the capacity of the jack or piston and the capacity and manufacture and welding of the apparatus components.

5.1.1 Paste rheology parameters and considerations:

The material viscosity has a governing influence on the resistance against extrusion flow. The high viscosity of the fibre-filled cement paste causes high shear and compressive forces. Cohesion forces exist within the matrix, which leads to a corresponding reaction force outwards, and in turn creating adhesion forces.

Bleeding under high pressure, the phenomenon when water is forced out of the mix, is another material characteristic that contributes to the increased flow resistance. It is common in extrusion of inorganic pastes to encounter a solid/ liquid phase separation during the paste flow in the extruder. This results in an increase of the extrusion pressure up to the die blockage. A suitably extrude-able mix is a mix that is of sufficiently high viscosity whereby, under high shear and compressive forces, orifice plugging, or the liquid 'sweating out' is prevented or limited. High viscosity is also required to prevent flow expansion and extrudate deformation due to rapid pressure reduction at the die outlet.

To alleviate the problems of high viscosity and solid/ liquid phase separation, a viscous agent is added. This is illustrated in Figure 26, where the influence of viscous agent (VA) on extrusion force is displayed. A balance between large enough force to improve the extrudate density and fibre orientation, and small enough force to avoid segregation, must be obtained. This led to the choice of a VA content of 1% for the extrusion mix, as described in section 4.1.1.2.

Fibre resistance against flow of the mix also depends on the type of fibre used, for example the mix resistance against flow for a steel fibre mix is more than that of a PVA fibre mix if all the other constituents are kept the same.

Aggregate grading also influences the paste rheology. The courser the aggregate grading, the more resistance to extrusion flow is generated by the mix.

Furhter research can be done on the sensitivity of the mechanical properties of the extrudates, as well as continuity and surface quality to the paste rheology parameters.

5.1.2 Extruder parameters and design concepts:

5.1.2.1 Transition of the extruder:

The die design for a specific geometry should fulfil constraints such as uniform flow, which is governed by the transition slope. The aim is to prevent stagnant zones from forming, to prevent texture change of the matrix being extruded and to combat flow instability that may all produce surface defects.

The piston-type extruder makes use of the wall friction and the reduction of the cross-sectional area from the loading chamber to the die, causing resistance to the flow of materials through the die. The greater the transition length between the ram section and the die section, the larger the total shearing resistance becomes.

The angle that the transition makes with the barrel axis changes the direction of reaction force acting on the material, hence influencing the normal forces acting on it. Too steep an angle could cause blocking of the extrudate at the front of the transition or where the die opening starts. Extrudates with a certain composite strength and shape are thus obtained. The critical angle θ_c is the maximum angle of a slope along which an ECC can be pushed forward under a horizontal force. A die angle larger than the critical angle is to be avoided since it causes no motion of the cement paste particles in contact with the interior wall of the die, no matter how large the applied force. In this case, the only possible way particles can move through the die cone (transition) is when a very high ram pressure is used to overcome the shear strength inside the cement paste. In this case a cone shaped shear failure plane develops and the paste inside this failure plane or cone will move through the die while a static zone of cement paste remains outside the failure cone (Figure 35). Therefore, when $\theta > \theta_c$, either the material clogs in the extruder when the ram pressure is not high enough or a very high ram pressure is needed to move the material through the extruder. The high pressure required not only wastes energy, but can damage the wall.

The limit on the die slope can explain certain extrusion phenomena. For example, a rough die (η is large and θ_c is small) is easy to clog since θ may be larger than θ_c. However, when the die interior wall is polished and/ or lubricant is applied, η decreases and θ_c increases. Thus, θ may become less than θ_c, and extrusion becomes possible at reasonable ram pressures. A large die angle means a steep slope of the die curve, which causes the die to clog easily.

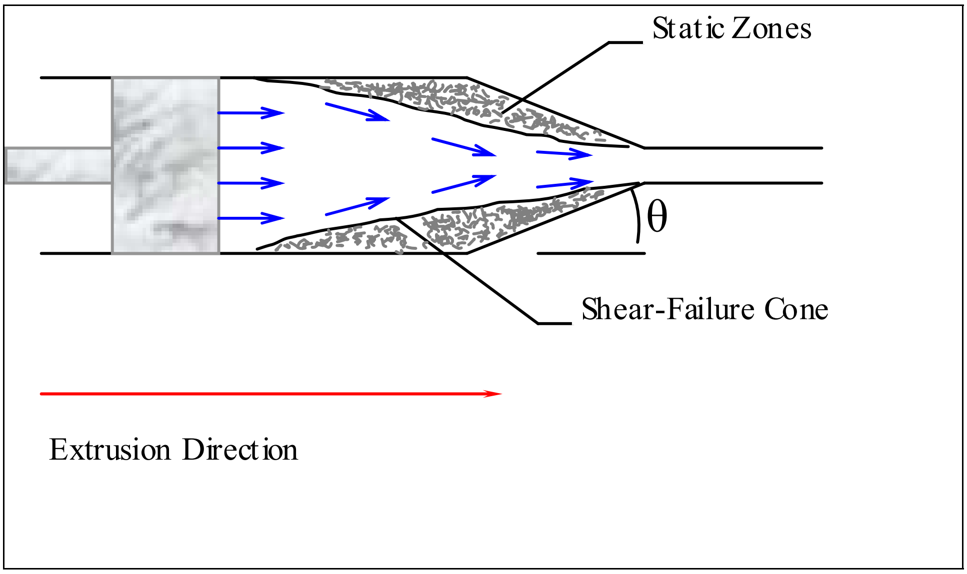


Figure 35: Static zone forming when $\theta>\theta_c$ (Shah et al., 1999)

5.1.2.2 Die shape:

Another parameter which has an effect on piston extrusion and on the extrusion load is the die shape. The extrusion pressure required in the paste extrusion process generally concerns the pressure drops along the die entry and the die-land. One for this is the larger product or extrudate surface to volume ratio, whereby total frictional resistance is increased. Another cause may be different transition gradients required by intricate die shapes, whereby overall resistance is increased. The die shape of the pipe piston extruder for instance could lead to a larger resistance.

It is known that once the pressure drop along the die has been determined, estimation of the flow characteristics of the paste is possible. Understanding the nature of the flow in dies is essential in order to control the extruder performance and extrudate quality. Inadequate control of the extrusion pressure causes liquid migration and die-blocking problems.

Analysis of the variation of pressure in the die helps to understand how the die geometry and the characteristics of the extruding material (powder/ paste) affect the extrusion load. In addition, a die designed for a certain material may not be suitable for extruding other materials because the characteristics of the extruding material determine the die design.

5.1.2.3 Compaction pressure in the barrel

As outlined above, the maximum compaction pressure that can be produced depends on the frictional resistance of the extruder wall to the column of compressed material, and on the counter-pressure acting on the material caused by the changing cross-section. The frictional resistance is determined by the radial pressure P_r acting on the interior wall of the die, by the coefficient of friction η, and by the area of the die wall. The counter-pressure of the wall for a conical die is mainly determined by the cone angle.

5.1.2.4 Piston (jack; driving mechanism):

The extruder driving mechanism must have sufficient capacity to extrude. Indications are that an extrusion pressure of up to 4 MPa may be required for ECC in the barrel (Shah et al., 1999). Provision for 10 MPa was made in the extrusion facilities built for this research at the University of Stellenbosch. Predetermination of the extrusion load is important for extruder design and application. For this reason, a load cell was implemented in the rheometer. However, at present, accurate determination of the extrusion load and the pressure variation in various sections of the die is not yet possible. To ascertain design parameters for designing the extruder, theoretical analysis and prediction of paste rheology as well as the interaction thereof with the extruder are required.

Through the piston rheometer tests, described in section 4.1.1, a mix design was obtained which provided an indication of the extrusion force required, albeit for the small scale piston extruder. This information was employed to estimate the extrusion forces and required strength of a larger piston extruder facility.

Furthermore, the influence of ram and die sectional area ratio and transition lengths on the internal pressure variations, as well as on the subsequent extrudate properties in terms of densification and fibre orientation, could not be accurately established. Rather, a process of trial and error was adopted by choosing these parameters by rough estimation, assuming Newtonian flow. The extrusion load depends on the magnitude of the total resistance in the die and transition where the raw material is compressed and densified. When the ram force supplied by the extrusion machine is high enough to overcome the total resistance of the die and transition, the compacted material is pushed forward and finally ejected from the exit of the die.

5.2 Piston extruder geometry and tests measurements

Keeping in mind the required design parameters outlined in 5.1, two sets of extrusion apparatus were designed and built in the Structures Laboratory of the Civil Engineering Department of the University of Stellenbosch.

One extruder was built for experimental plate extrusion for mechanical response characterisation. A separate, second extruder was built for pipe extrusion. This was done for ease and speed of fabrication of pipe products as well as sample plates for testing.

Figures 36-40 show various views of the plate and pipe extruders.

Figure 36: The pipe piston extruder (above left) and the plate piston extruder (above right)

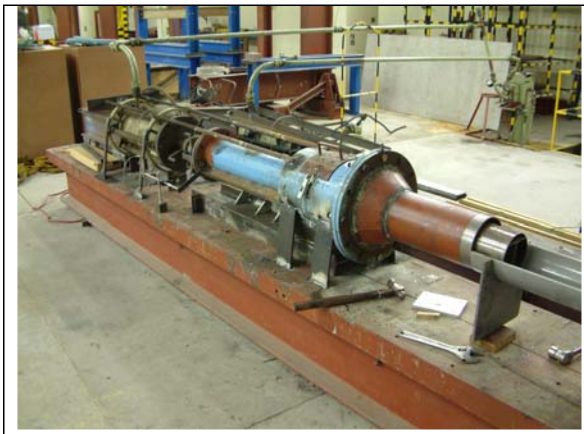


Figure 37: Pipe piston extruder

Figure 38: Plate piston extruder

Figure 39: Pipe piston extruder (front)

Figure 40: Plate piston extruder (front)

As discussed in chapter 3, the research focus was the determination of densification and fibre orientation caused by fabrication processes. It is recognised that the extruder geometry may have a significant influence on these two characteristics. In this research the sensitivity of these characteristics to the extruder geometries was not studied. However, the extrusion force, a major role player in both these two phenomena, was monitored during extrusion of both plate and pipe specimens. In Figures 41-44 the extruders and the measured extrusion force verses extrusion stroke are shown. Tables 11 and 12 list the extrusion parameters of the respective extruders.

Figure 41: Plate piston extrusion

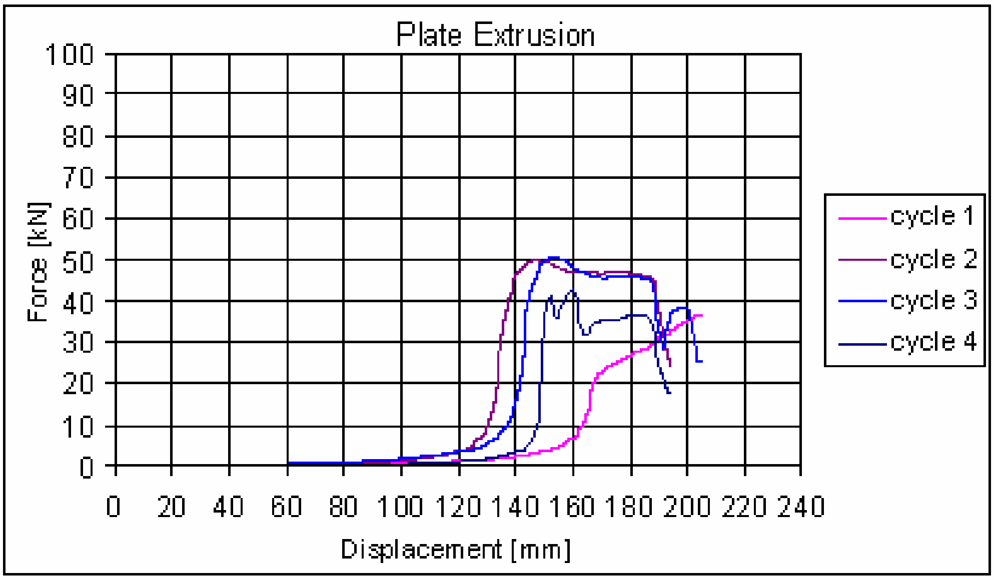


Figure 42: Force vs. Displacement graph for plate extruder piston cycles

Dimensions of the plate extruder barrel Dimensions of the plate extruder die Transition angles Die Cross-section: barrel cross-section ratio Maximum force Measured for specific mix	108mm x 144mm (x 300mm length) 70mm x 15mm (x 400mm length) 21° (side view) and 17° (top view) 0.0675 50 kN (≈3.2 MPa barrel pressure)
Average extrusion velocity	6.9mm/s
Average extrudate velocity	102.3mm/s

Table 11: Plate extruder parameters

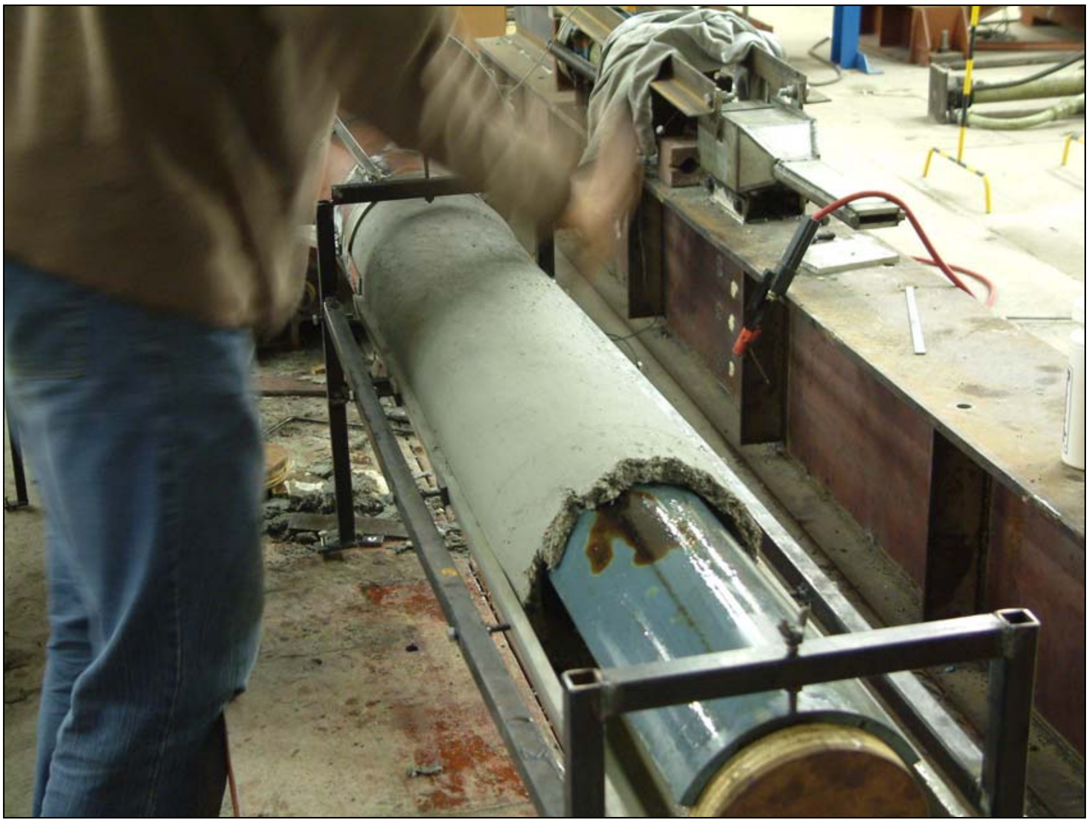


Figure 43: Pipe piston extrusion

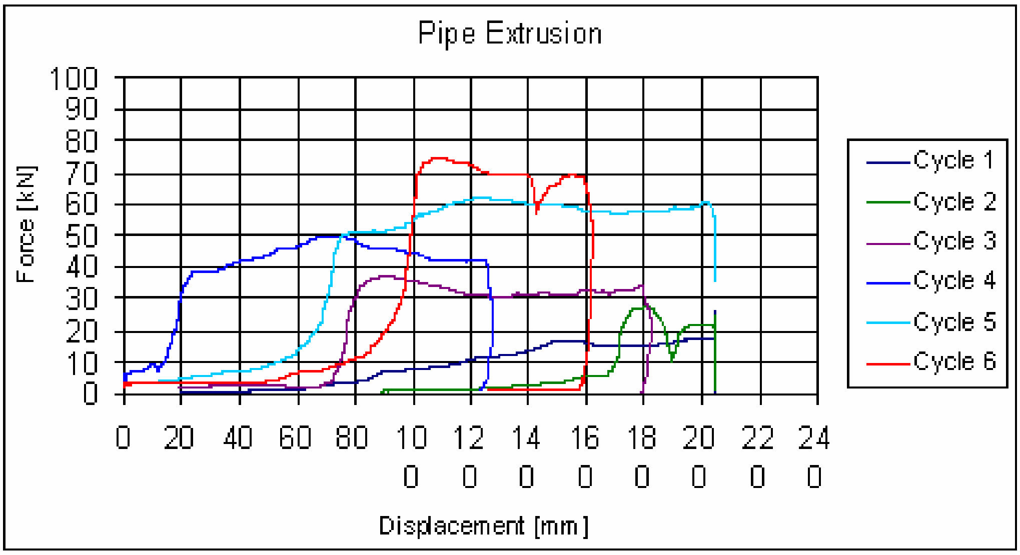


Figure 44: Force vs. Displacement graph for pipe extruder piston cycles

Dimensions of the pipe extruder barrel	300mm outer φ x 144mm inner φ (x 300mm length)
Dimensions of the pipe extruder die	180mm outer φ x 150mm inner φ (x 400mm length)
Transition angle	20°
Die cross-section: barrel cross-section ratio	0.0948
maximum force measured for specific mix	75 kN (≈1.4 MPa barrel pressure)
Average extrusion velocity	3.03 mm/s
Average extrudate velocity	20.65 mm/s

Table 12: Pipe extruder parameters

From the results it can be seen that reasonable agreement in extrusion forces between plate and pipe extruders are of the same order of magnitude, despite the different geometries. This is essential, to be able to use extrusion design parameters for future research.

With all other parameters being the same, matrix properties such as the tensile strength and densification are affected by the method of processing. Fibre orientation and some micro-mechanical properties such as interfacial bond strength and first cracking strength are also affected by the manufacturing technique. This presents some difficulty in independent control of each micro-mechanical parameter during mix design. Even when the influence of the manufacturing technique on fibre orientation, fibre dispersion, interfacial bond strength and matrix densification are exactly quantified, it would still need extensive modelling to see the combined result on the matrix properties.

As a result additional experimental tests and interpretations were needed to ascertain the combined influence of cast and piston extrusion (respectively) on the coupled parameters. Figure 33 gives an outline of the testing program that was followed.

Cast Composites	**Piston Extruded Composites**	***Ref***
Compression tests: cubes	-	*§6.1*
Tensile tests: dog-bones	-	*§6.2*
Three point bending tests: plates	Three point bending tests: plates	*§6.3*
Crushing tests: pipes	Crushing tests: pipes	*§6.4*
Flexural (three point bending) tests: pipes	Flexural (three point bending) tests: pipes	*§6.5*

↓

Cast Composites vs. Extruded Composites	
Three point bending tests: plates	*§6.3*
Crushing tests: pipes	*§6.4*
Flexural (three point bending) tests: pipes	*§6.5*
Densification/ compaction	*§6.6*

Figure 45: Outline of testing program

6.1 Compressive strength

At 21 day age, the compressive strengths of the cast composite mix are listed in table 13.

Measure1	Measure2	Measure3	Average	Stand.Dev.	COV
52.0 MPa	52.0 MPa	53.5 MPa	52.5 MPa	0.87	0.02

Table 13: Compression test results of the cast composite specimens

It can be postulated that the compressive strength of the cast composite mix used in this research is rather low compared with other tailored cast composite mixes developed by a parallel research group at the University of Stellenbosch, where the compressive strength enhancement by steel fibre of up to 150% has been recorded (Song and Van Zijl, 2004).

This is largely due to the high fly ash content (Table 8). Keep in mind that the water:cement ratio is in fact higher than indicated, due to the fact that much of the binder consisted of fly ash, of which the portion above 30-40% of the binder by mass does not participate in the hydration process.

Another possible reason for the relatively low compressive strength results could be due to a fibre induced damage effect to the matrix, which results in a composite with higher porosity compared with the matrix material alone (Li et. al., 1995). This contradicts the compressive strength enhancement results mentioned above.

6.2 Tensile strength

Direct tension tests were conducted to evaluate the performance of the cast composite materials with the same matrix constituents as the extrusion composites, but with different quantities of chemical additives additives to enable manufacture of the specimens, as discussed in 3.1.3.

The tensile stress-strain curves of the three cast specimens are shown in Figures 47 and 48. The matrix exhibited typical strain-softening behaviour in the case of two samples, whereas the third sample exhibited a strain hardening type of response. All the samples exhibited single matrix cracking domination, a typical quasi-brittle response characteristic (Figure 46).

Figure 46: Tensile test specimens with single matrix cracking domination

The average peak tensile parameters for the three tensile test samples are listed in table 14 below.

	Average	Standerd Deviation	Coefficient of Variance (%)
Tensile strength:	5.57 MPa	0.54	9.75
Strain:	1.90 %	0.20	10.37

Table 14: Average peak tensile parameters

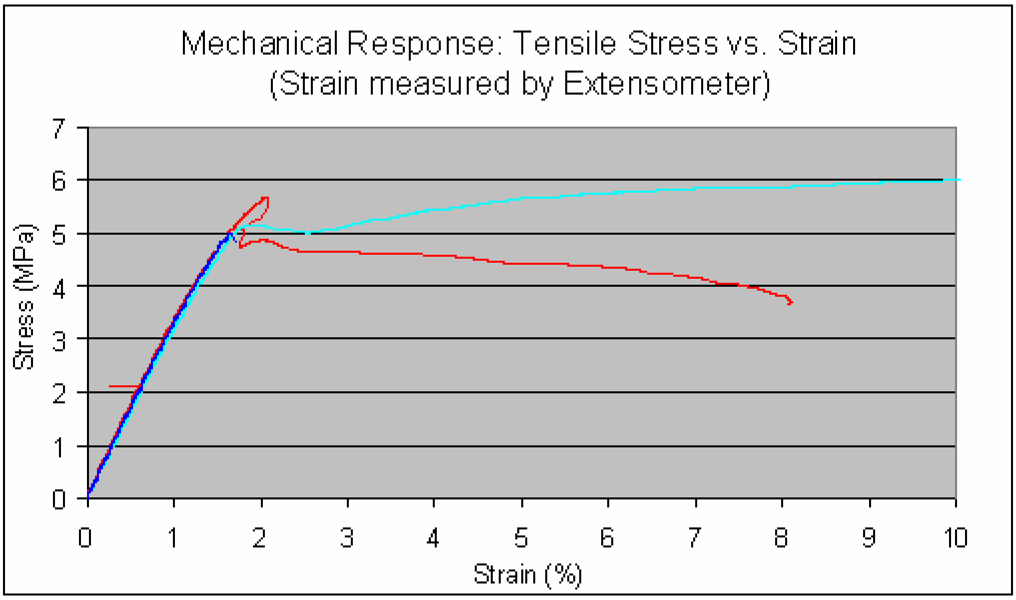


Figure 47: Typical stress-strain curves of cast steel fibre composites (strain measured with extensometer)

It should be noted that the strain beyond the peak in Figure 47 was measured incorrectly by the extensometer. This was caused by skew cracking, running from within the gauge area diagonally to outside the gauge area. Thereby, the extensometer was somehow corrupted, perhaps through slipping of the extensometer gauge tips into the crack that developed where cross-section widening started. One recommendation that can be drawn from our experience is that LVDT measurements should be conducted in the future.

Due to the incorrect extensometer post-peak measurements, an approximation of the strain was calculated using the increase in distance between the Zwick clamps, i.e. the built in Zwick displacement meter (Figure 48). These measurements are merely an approximation of the ductile behaviour of the composites, and reflect a more brittle response.

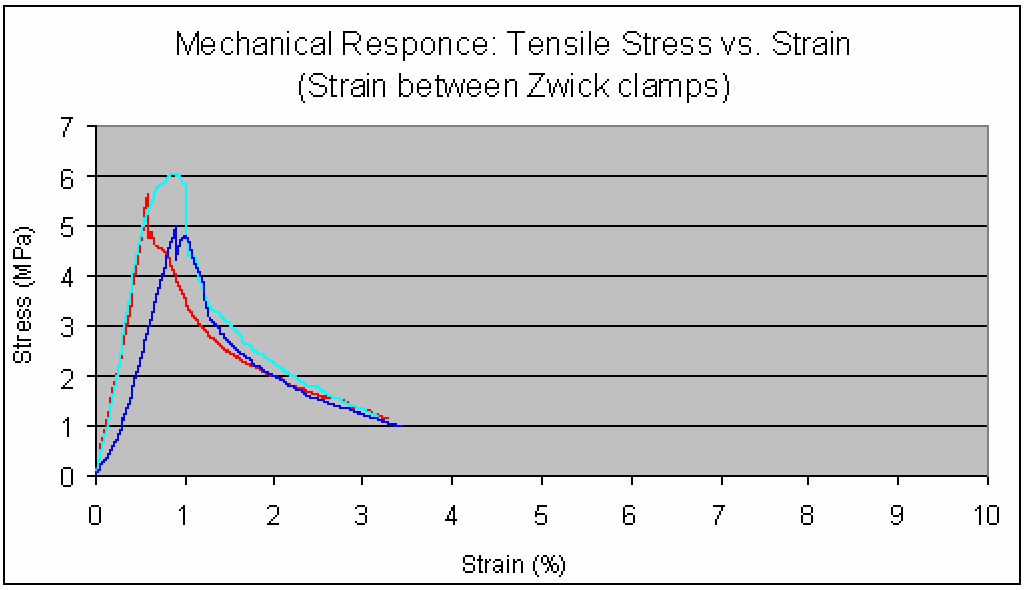


Figure 48: Typical stress-strain curves of cast steel fibre composites - strain measured between the clamps of the materials testing machine (Zwick Z250)

6.3 Indirect tensile strength (three point bending)

Three point bending tests were conducted to determine what the influence of piston extrusion compared to casting was on the flexural performance. Bending stress-deflection curves (Figure 49) and force deflection curves (Figure 50) demonstrate the significant increase in the flexural strength behaviour of extruded composites. As would be expected, the extruded plate composites not only exhibit a higher peak flexural strength, but it also had more ductility even though quasi-brittle response prevailed.

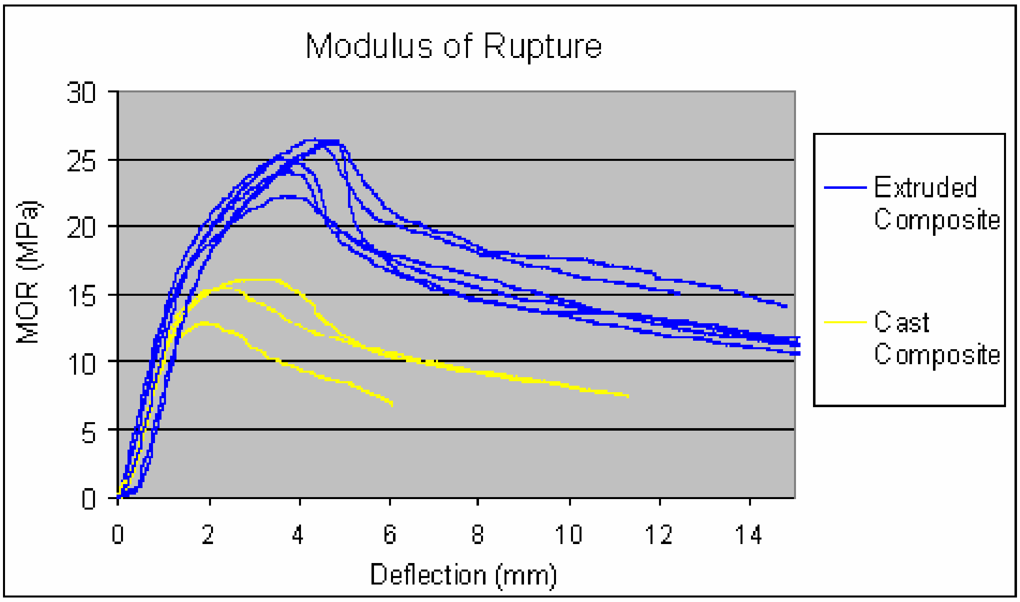


Figure 49: Bending stress-deflection curves of extruded and cast steel fibre composites

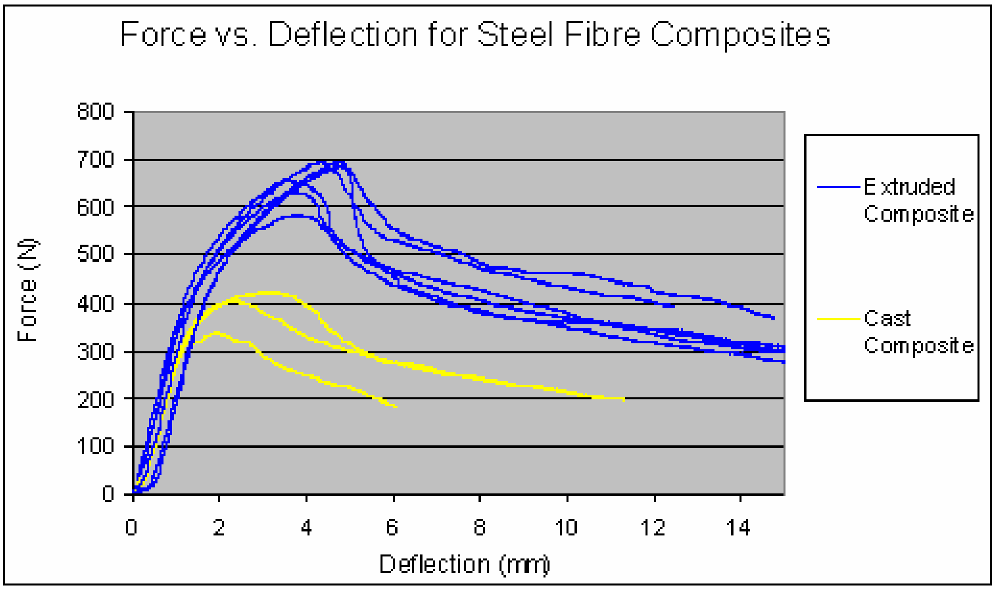


Figure 50: Bending force-deflection curves of extruded and cast steel fibre composites

When subjected to high stress gradients, three-point bending loading produces stress gradients and reveals that the tough uni-axial tensile response may cause a MOR of several times the composite tensile strength. However, this cannot be achieved by the less ductile

matrix, as tensile softening degradation upon higher straining of fibres far from the neutral axis limits the ultimate resistance in flexure.
The cast SFRC results in Table 15, Figures 51 & 52 show average MOR values of 2.7 times that of the average tensile strength.

The average peak tensile parameters for the three tensile test samples and three flexural test samples (three point bending samples) are listed in table 15 below.

	Average	Standerd Deviation	Coefficient of Variance (%)
Three point bending results	14.9 MPa	1.5	10.2
Tensile results	5.6 MPa	0.5	9.7

Table 15: Average peak tensile and flexural parameters

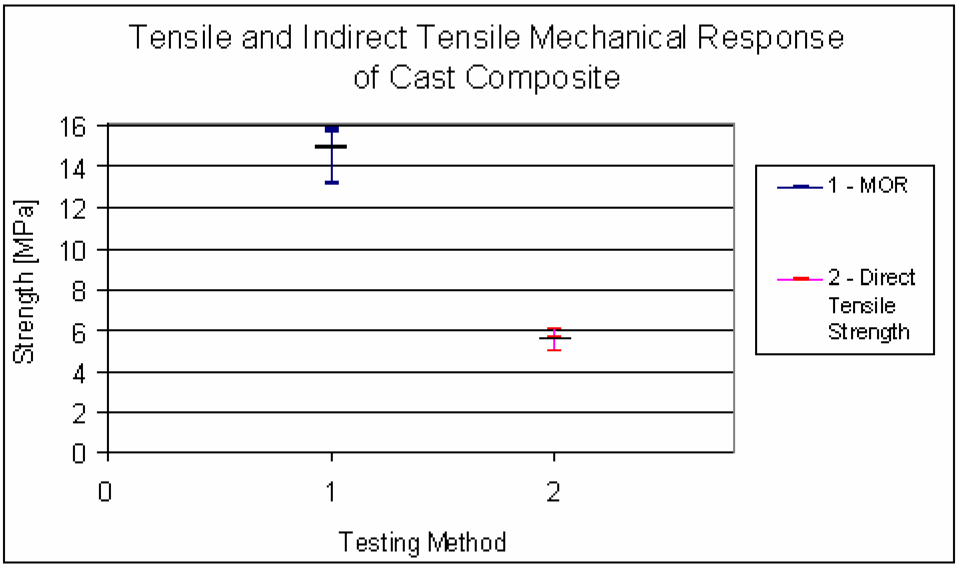


Figure 51: Tensile vs. Indirect tensile mechanical response of cast SFRC

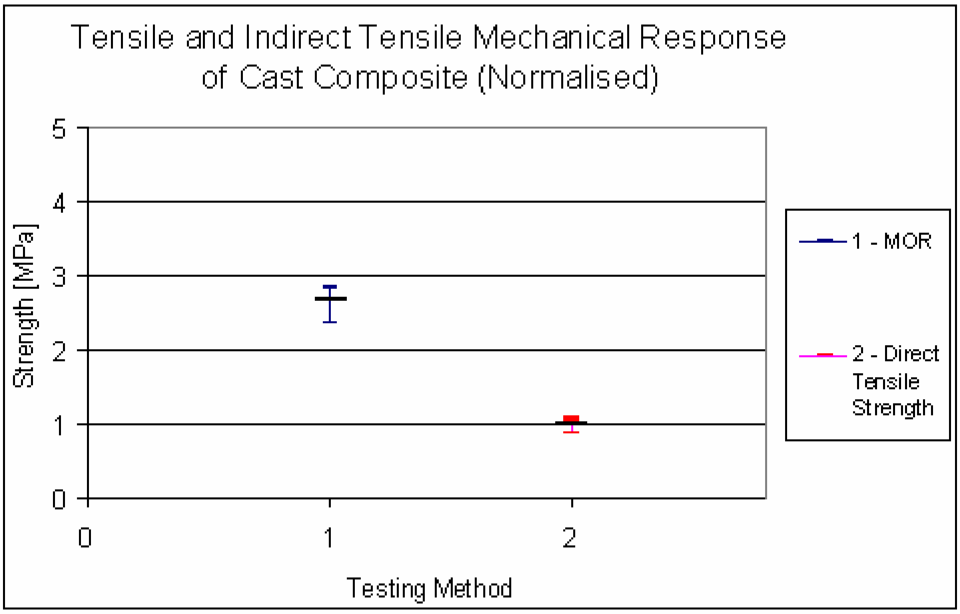


Figure 52: Normalised results of tensile vs. indirect tensile mechanical response of cast SFRC

6.4 Pipe crushing tests

Figures 53 and 54 present behaviour of two manufactured piston extruded pipes, called piston pipe 1 and piston pipe 2. An overall look at the results indicates a poor performance in crushing strength of piston extruded SFRC pipes.

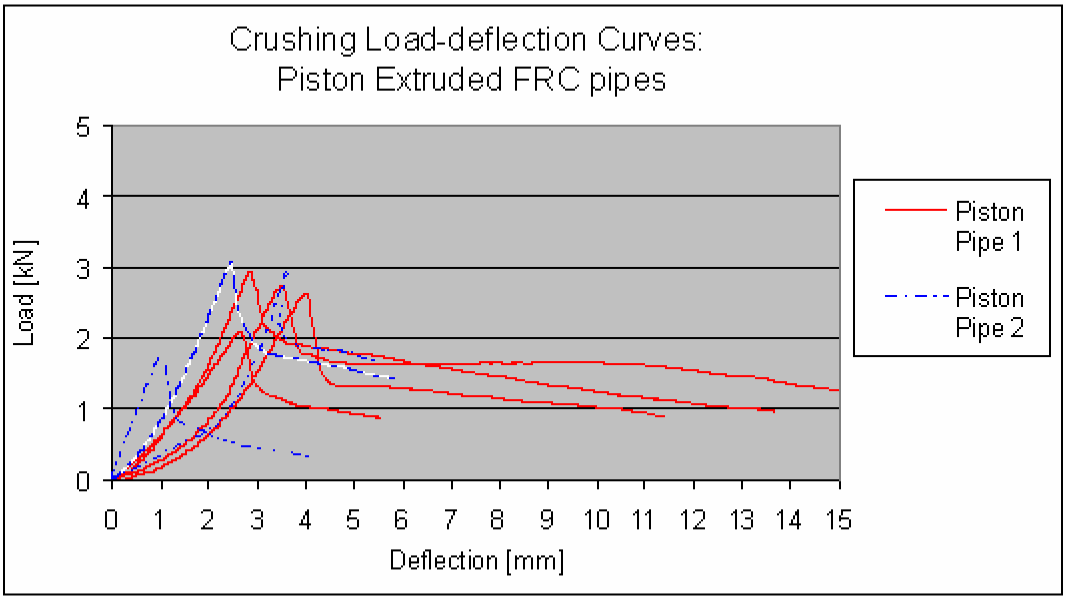


Figure 53: Crushing load vs. deflection curves for piston extruded SFRC pipes

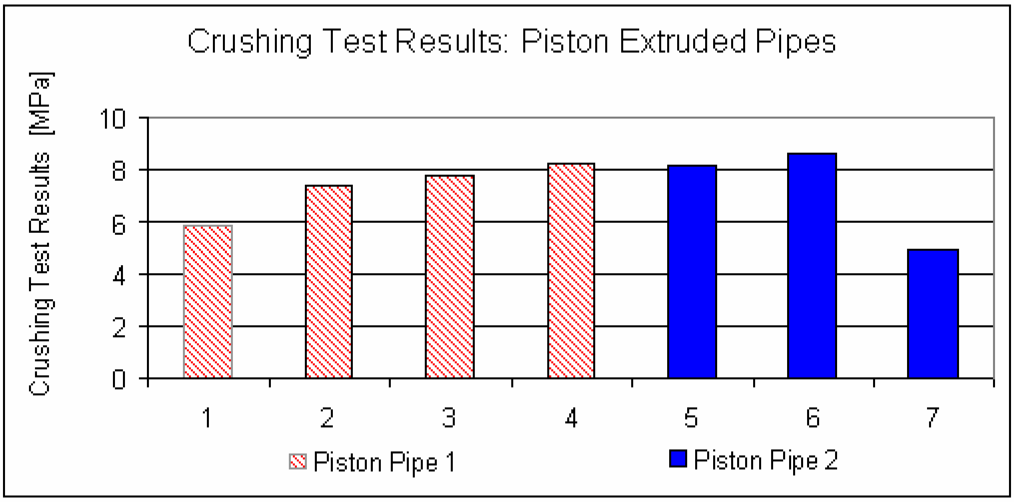


Figure 54: Crushing strength results for piston extruded SFRC pipes

Several cracks formed along the length of the pipe test specimens. This is due to anisotropic behaviour of the pipes due to the fibre orientation only in the longitudinal direction, leaving insignificant transverse reinforcing (Figure 55). This resulted in the cracks developing longitudinally between and parallel with the fibres.

Figure 55: Anisotropic behaviour of piston extruded pipes in crushing tests

The crushing load-deflection behaviour of the cast SFRC pipes displayed higher crushing strength results. Figures 56 and 57 present the results of two manufactured cast pipes, called cast pipe 1 and cast pipe 2.

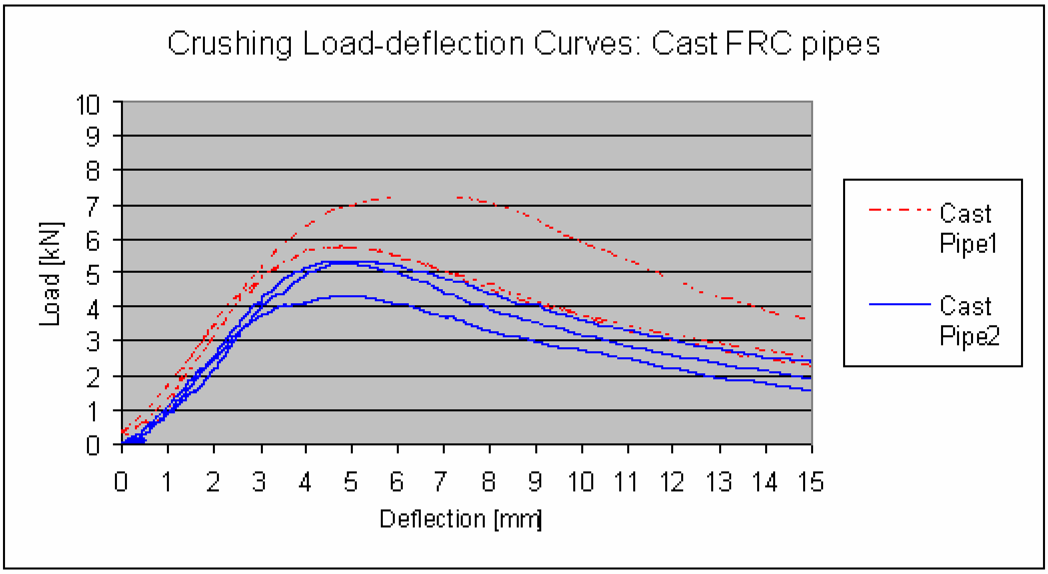


Figure 56: Crushing load vs. deflection curves for cast SFRC pipes

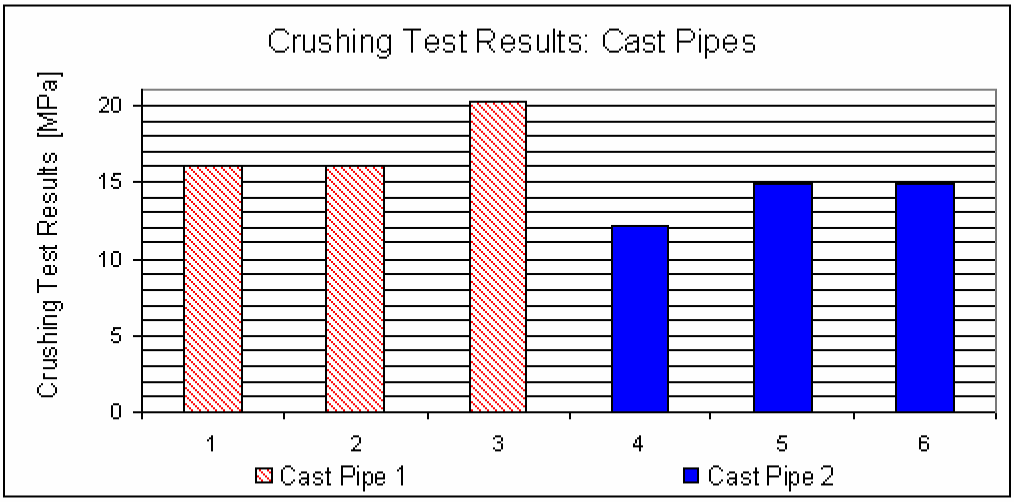


Figure 57: Crushing strength results for cast SFRC pipes

The pipes were thin shell sections, with a thickness-to-nominal diameter ratio of 1:10. The thin shell effect contributed greatly to the contradicting lower anisotropic crushing response of the pipe sections compared with the plate specimens discussed in 6.3, and distinguish the optimisation techniques for the two respective cross-sectional shapes (Figures 58 & 59 have reference).

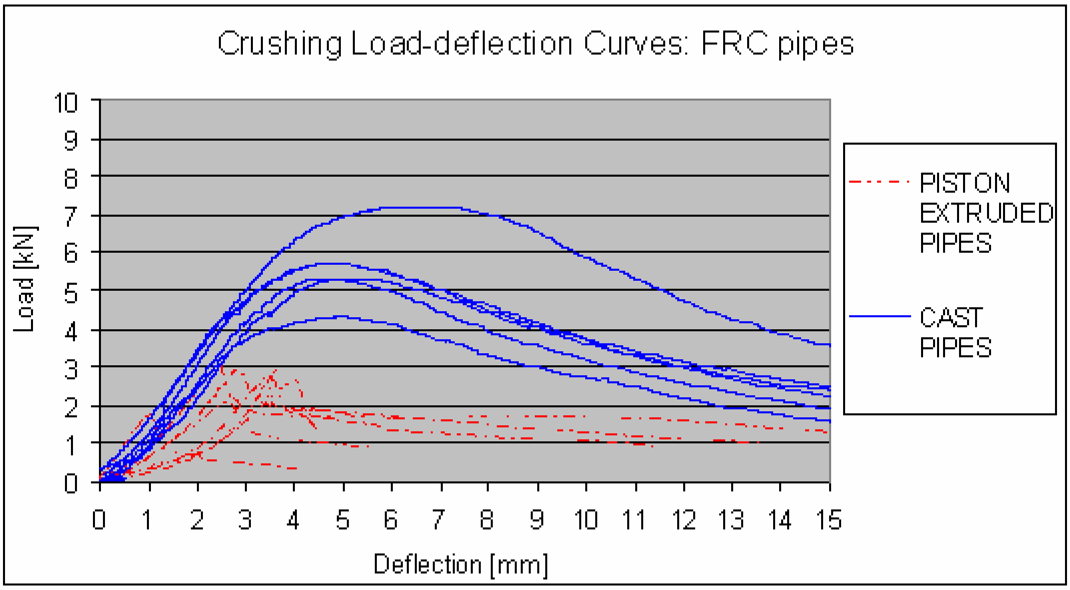


Figure 58: Crushing load vs. deflection curves comparing cast and extruded SFRC pipes

The steel fibres increase the flexural strength of the ECC in comparison with unreinforced ECC. For thin wall pipes, the fibre reinforcement was needed in both the longitudinal direction and the circumferential direction because they undergo cross-sectional ovalling under overload. The steel fibre reinforced pipes still exhibited distinct plastic behaviour before failure. The incorporation of steel fibres in the ECC increased the strength of the pipes significantly.

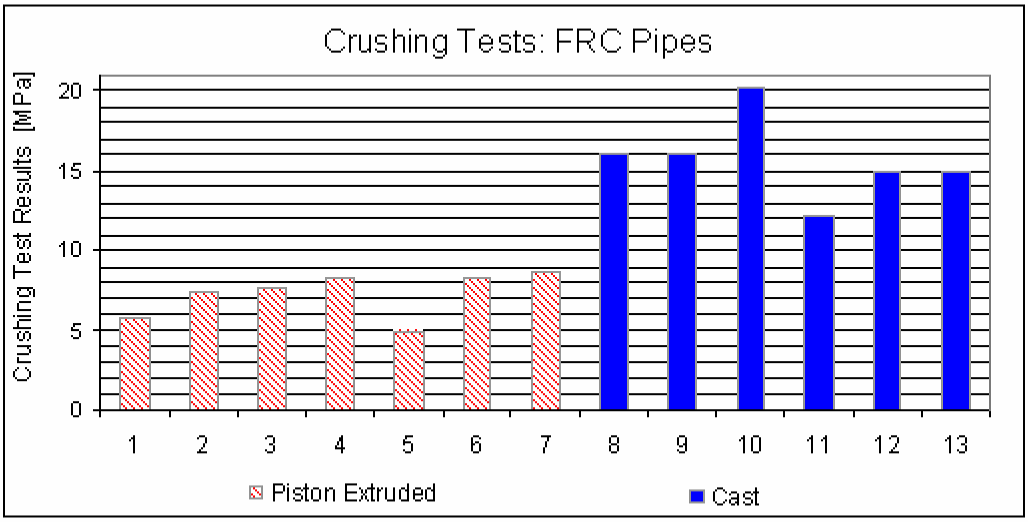


Figure 59: Crushing strength results for extruded and cast SFRC pipes

6.5 Flexural tests (three point bending – pipes)

Flexural tests on SFRC pipes were only performed on piston extruded specimens. Cracks along two generating lines at the ends of a diameter normal to the loading plane were noticed at failure (Figure 60).

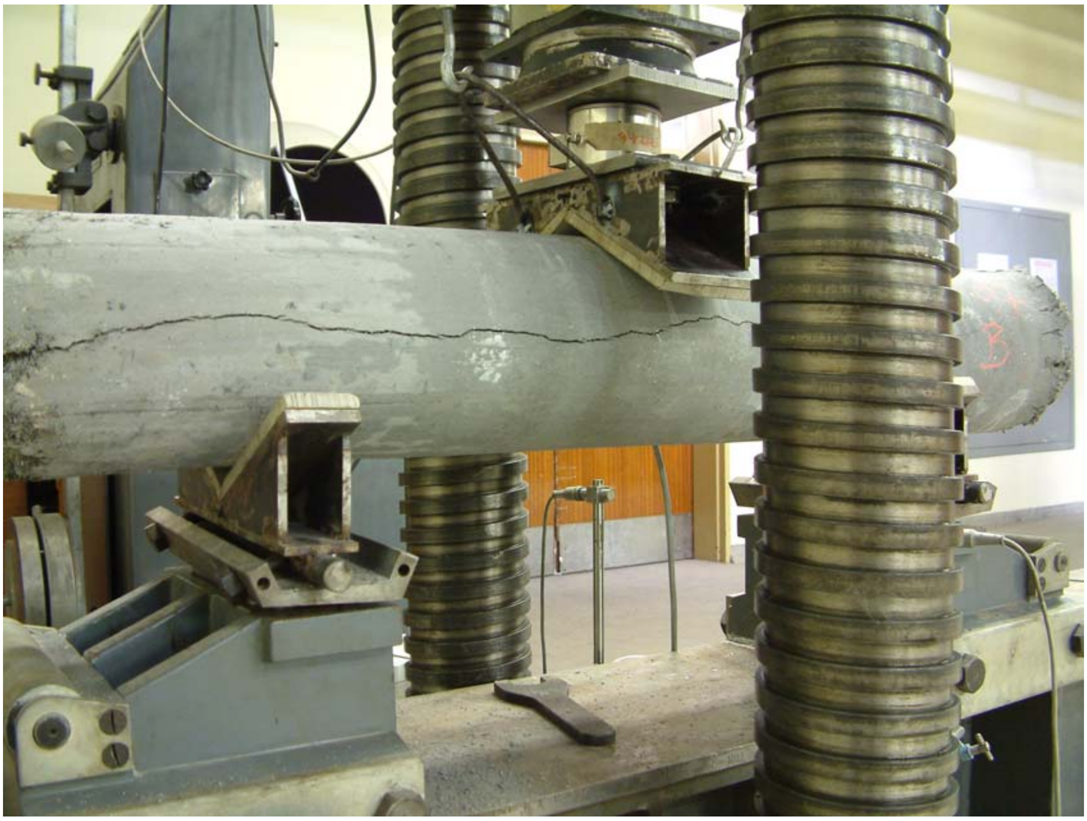


Figure 60: Anisotropic behaviour of piston extruded pipes in flexural tests

Figure 61 and 62 present the flexural behaviour for seven piston extruded SFRC pipes. The first three are called piston pipe 1, two more pipes are called piston pipe 2 and the last two pipes are called piston pipe 3. The graphs indicate that the performance in flexural strength of extruded SFRC pipes was low and that anisotropic behaviour prevailed as was the case with the crushing tests.

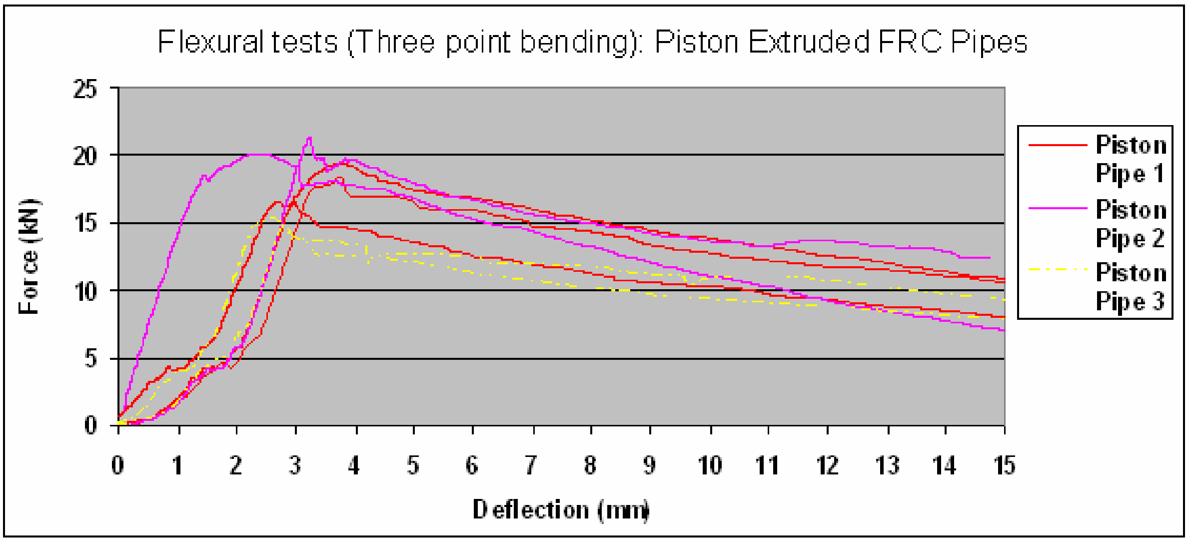


Figure 61: Force vs. deflection behaviour of piston extruded SFRC pipes

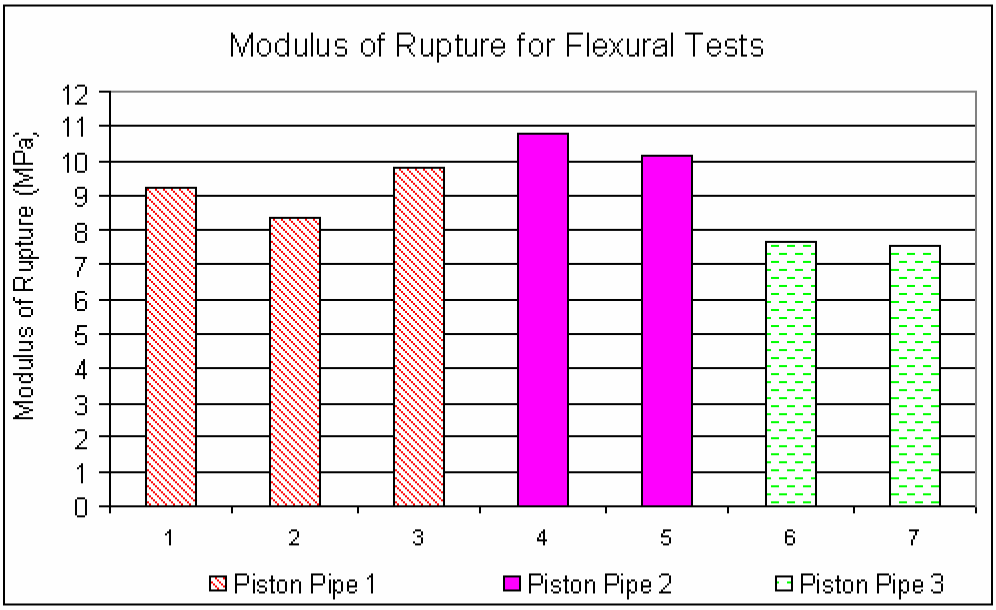


Figure 62: MOR test results of piston extruded SFRC pipes

6.6 Densification/ compaction

Another property that is influenced by the manufacturing method is the density of the composite. The method of extrusion and the design features of the specific extruder did not only determine the fibre orientation, but also provided mechanical compaction.

It can be postulated that an increase in density is coupled with an increase in interfacial bond strength of the composite, hence increasing mechanical properties such as composite strength and matrix toughness of the fibre composite in the direction of the aligned fibres.

However, with all the other parameters being the same, the lower the matrix toughness, the easier it is to achieve ductile behaviour of the composite. This situation places contradicting demands on the extrusion process: the extrusion process leads to a higher density of the composite creating more brittle behaviour, yet it creates a desired fibre orientation.

Different density values for cast composites and piston extruded composites for plate and pipe specimens respectively, are shown in Figure 63. These specimens were taken from the various specimens tested 6.2 – 6.5.

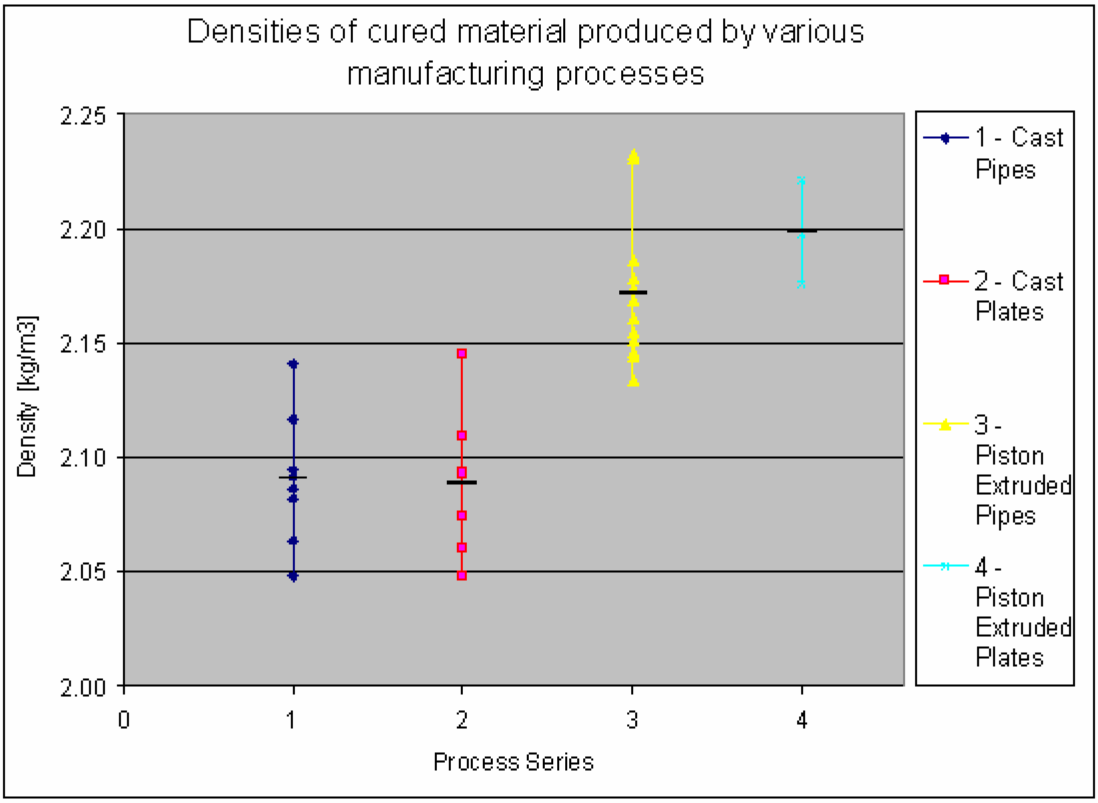


Figure 63: Influence of manufacturing processes on density

The design features of the extruder such as the transition angle, die length, extrusion speed and the 'barrel: die area' ratio led to higher densification of the extrudate.

7. Fibre Orientation

ECC may be manufactured by different manufacturing processes: cast moulding, piston extrusion and auger extrusion. Specimens or products of the different processes exhibit different fibre orientations.

7.1 Fibre orientation in extruded and cast specimens

The interaction between individual fibres influences the orientation of fibres. In the process of developing optimum mix designs for extrusion composites, there was a problem with fibre clustering in the case of the longer fibre lengths. This would undoubtedly have an effect on the fibre orientation. The fibre orientation is hence optimized using shorter fibres during the extrusion process. This is just one of the contradictory trends observed when comparing the mechanical behaviour of cast and extruded composites (Shah et. al., 2003).

Comparing the microstructures, Figures 64 and 65 show SEM observations of fibre dispersion for cast and piston extruded composites respectively. Although difficult to clearly substantiate from these images, it was observed that the extruded composite had more fibres aligned in the direction of extrusion. The fibre orientation of the extruded composite of Figure 65 is expected to have a greater influence on the mechanical properties than the cast composite in Figure 64.

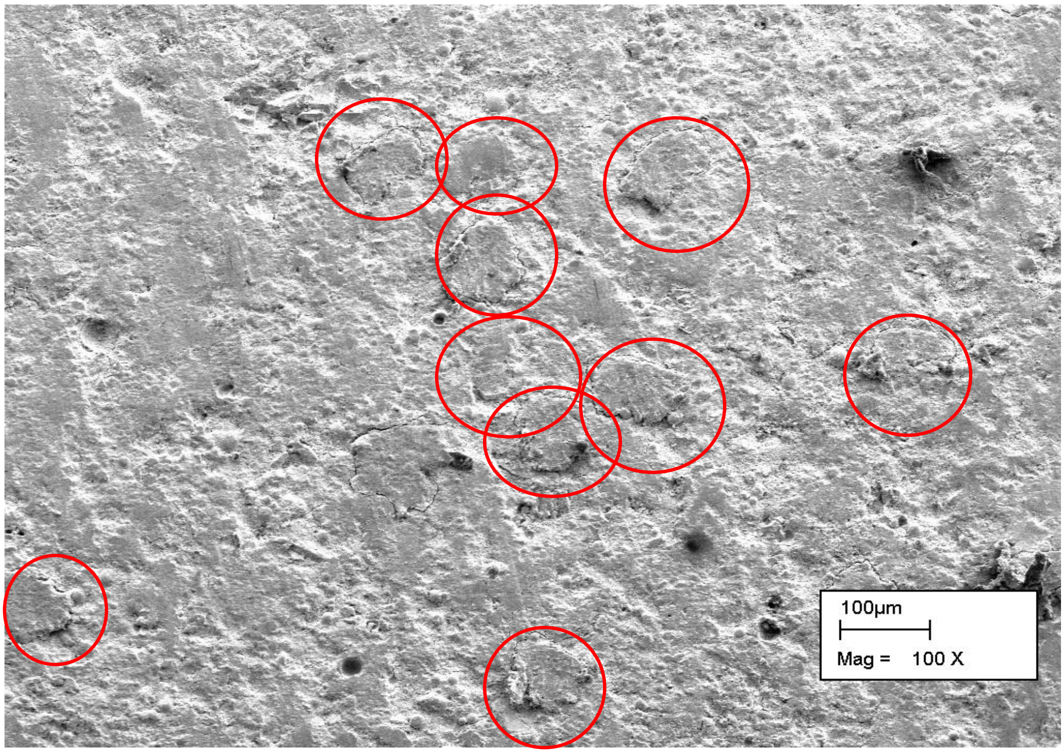


Figure 64: SEM image of cast composite

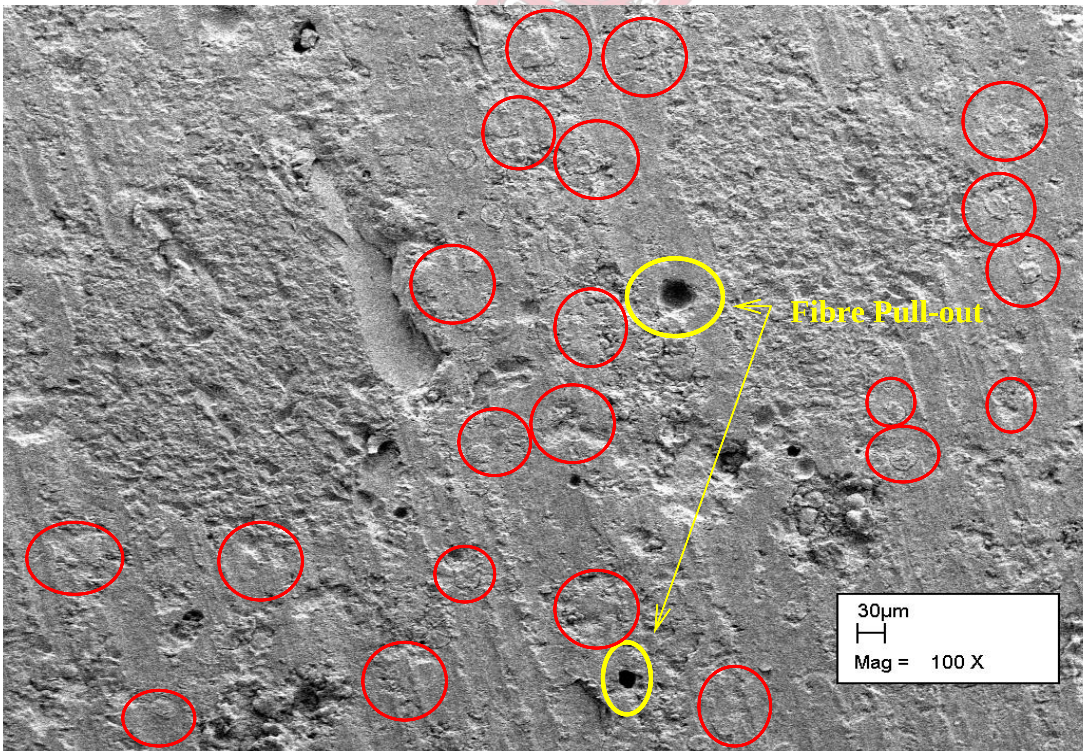


Figure 65: SEM image of extruded composite

To clearly differentiate between the influence of the different manufacturing processes on the fibre orientation, CT scans were performed on auger extruded plates (Figure 66), auger extruded pipes (Figure 67), piston extruded plates (figure 68), piston extruded pipes (Figure 69), cast plates (Figure 70), cast pipes (Figure 71) and spinned pipes (refer Figure 72).

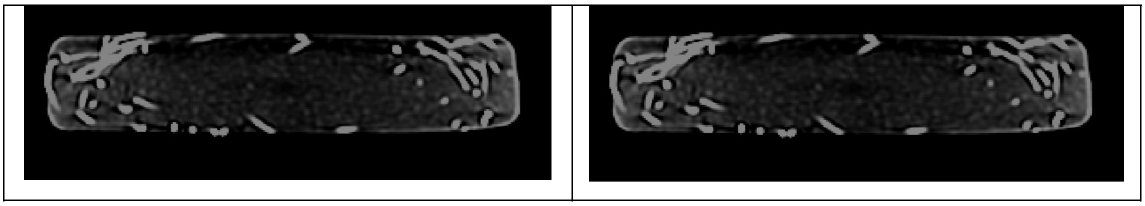


Figure 66: CT scan images of auger extruded steel fibre reinforced ECC plate

Figure 67: CT scan images of auger extruded steel fibre reinforced ECC pipe

In extruded products the fibres are directed in the load-bearing direction of the mechanism driving it. A marked tendency was observed of fibres to orientate in the axial direction in piston extrusion (Figures 68 and 69) and in the helix pattern of the auger driving gear (Figures 66 and 67), as opposed to random orientation in cast specimens.

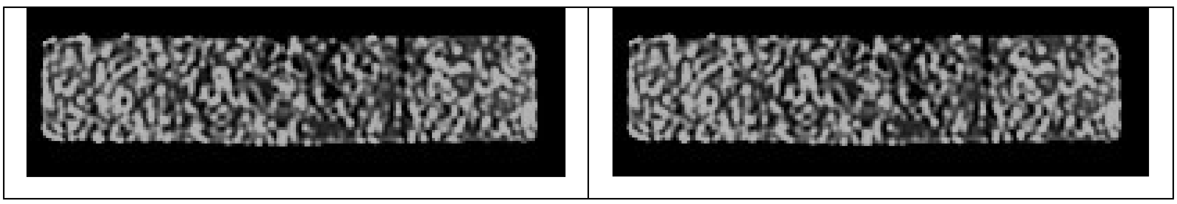


Figure 68: CT scan images of piston extruded steel fibre reinforced ECC plate

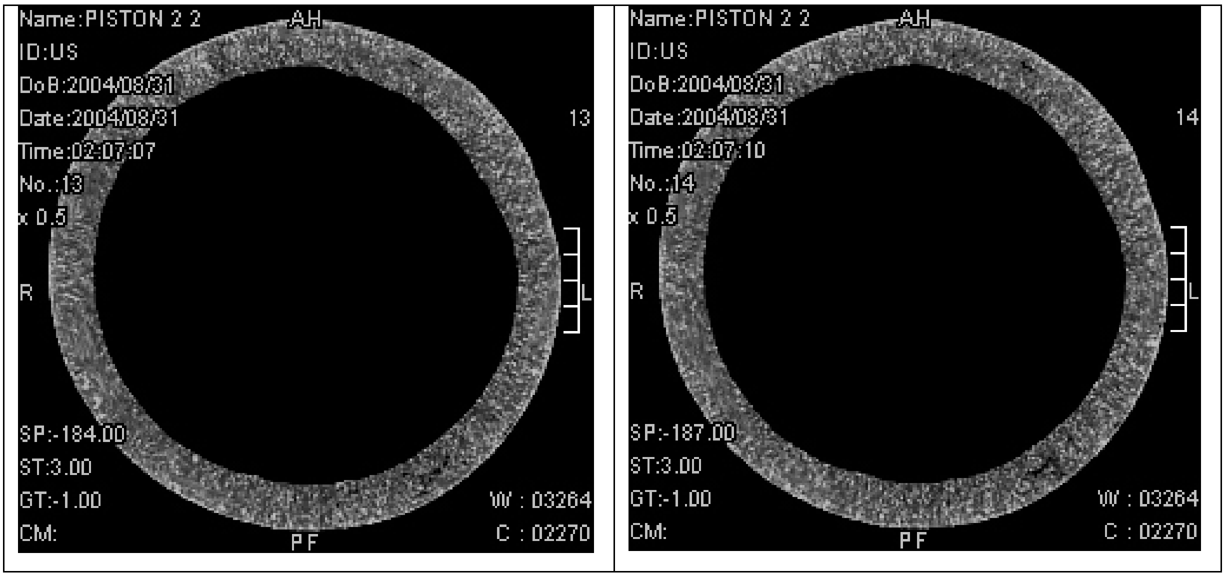


Figure 69: CT scan images of piston extruded steel fibre reinforced ECC pipe

Cast specimens exhibit random distribution of fibres, except near boundaries. Figures 70 and 71 indicate a random fibre orientation CT scan images and also show no fibres segregating to the bottom when it is vibrated.

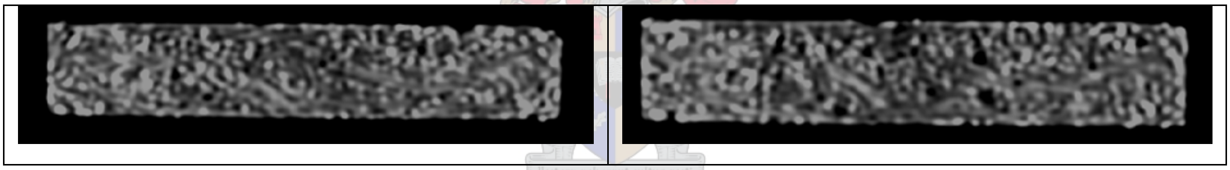


Figure 70: CT scan images of cast steel fibre reinforced ECC plate

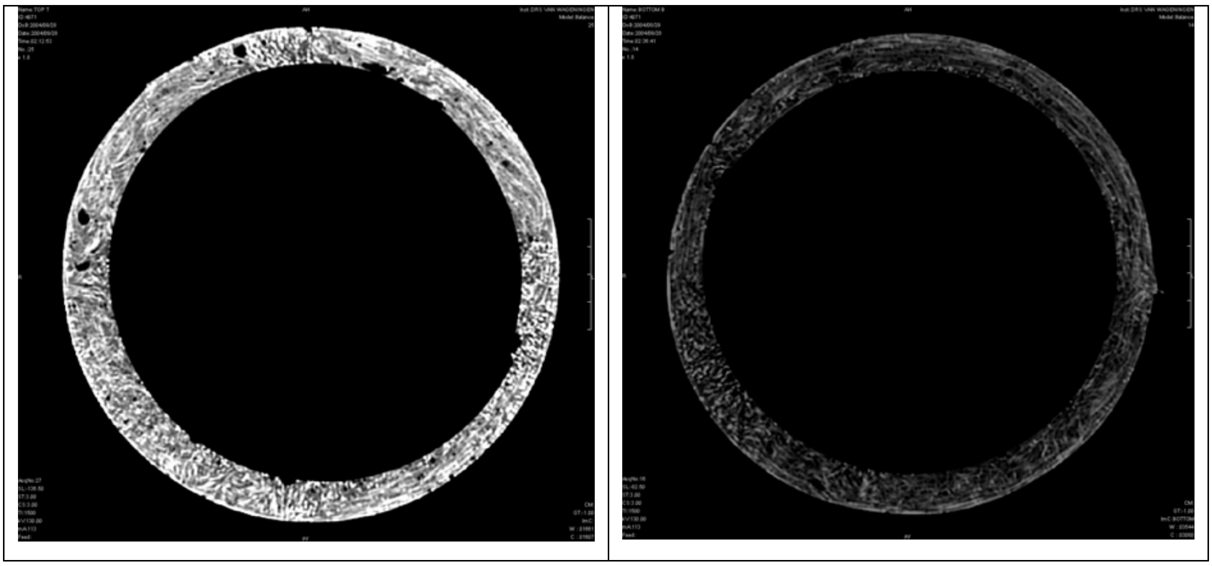


Figure 71: CT scan images of cast steel fibre reinforced ECC pipe

Test results coupled with fibre orientation (for cast and piston extruded composites)

In addition to the CT scans, the test results in Chapter 6 are indicative of the difference in mechanical response for cast and piston extruded composites.

In 6.2 the tensile test results for the cast composites display a ductile quasi-brittle response. The cast composite tensile test specimens displayed single matrix cracking domination. No direct tensile tests were performed on extruded specimens, because dog-bone shaped specimens suitable for such tests could not be extruded and were also not machined from extruded plates. In predicting the direct tensile response of extrudates, the contradicting influences of fibre snubbing (enlarged pull-out resistance due to non-alignment of fibres) versus the larger number of fibres crossing the cracks by the extrusion alignment, cause a predicament. It can only be postulated that the mechanical response of piston extruded samples could generate a lower tensile strength mechanical response due to fibre alignment in the longitudinal direction coupled with less fibre snubbing, whereas the ductility is increased due to more fibres bridging the cracks. It could further be postulated that the tensile response would remain typically quasi-brittle since the mode of failure remains fibre pull-out. To shed light on this issue, the indirect tension test results are discussed next.

The three point bending tests in section 6.3 revealed that the tough uni-axial tensile response for the cast composites produced a MOR of 2.7 times the average tensile strength. This can only be obtained with a more ductile matrix. It could be postulated that the MOR: tensile strength ratio would be greater for piston extruded composites since more matrix ductility is produced by the fibre alignment. The superior mechanical response of the extruded plates is clear from the comparison of flexural results with those of cast plates. This is an indication of the benefit of fibre orientation parallel to the load resisting action and also indicates dominance of increased number of fibres bridging cracks in extrudates above the effect of the fibre snubbing factor acting in cast specimens.

The cast SFRC tensile test and MOR results in Figures 51 and 52 are much lower than PVA tailored mixes which are more ductile and produce MOR: tensile strength ratios exceeding 5.0 (Song and Van Zijl, 2004).

The better crushing load-deflection performance of the cast pipes over the piston extruded pipes (Figures 58 and 59) is due to anisotropic behaviour of the extrudates. Unlike the plate composites, the pipe sections need fibres in both the longitudinal and circumferential directions to resist the biaxial action.

7.2 Fibre orientation in spinned pipes

The process of pipe spinning tends to orientate the fibre in the circumferential direction. The spindle forces the fibre in the direction of the movement through a Coulomb friction action. This happens throughout the pipe wall as it is built up in the process of continuously increasing deposited material during spinning, increasing the thickness.

Figure 72: CT scan images of a spinning steel fibre reinforced ECC pipe

As an indication of the improved mechanical properties in the direction of fibre orientation, the responses to a crushing test of spinning pipe sections of ECC are compared with those of lightly steel reinforced concrete pipe sections (Figure 73). Note the enhanced performance of the ECC pipes in terms of pseudo strain hardening and post peak toughness. Note also that this is merely illustrative, as lightly reinforced concrete pipes of greater dimension than those of the ECC pipes, produced by spinning, are reflected in this figure. Attention is drawn to the more ductile response of the ECC pipes, in which the fibres are orientated circumferentially through the spinning process.

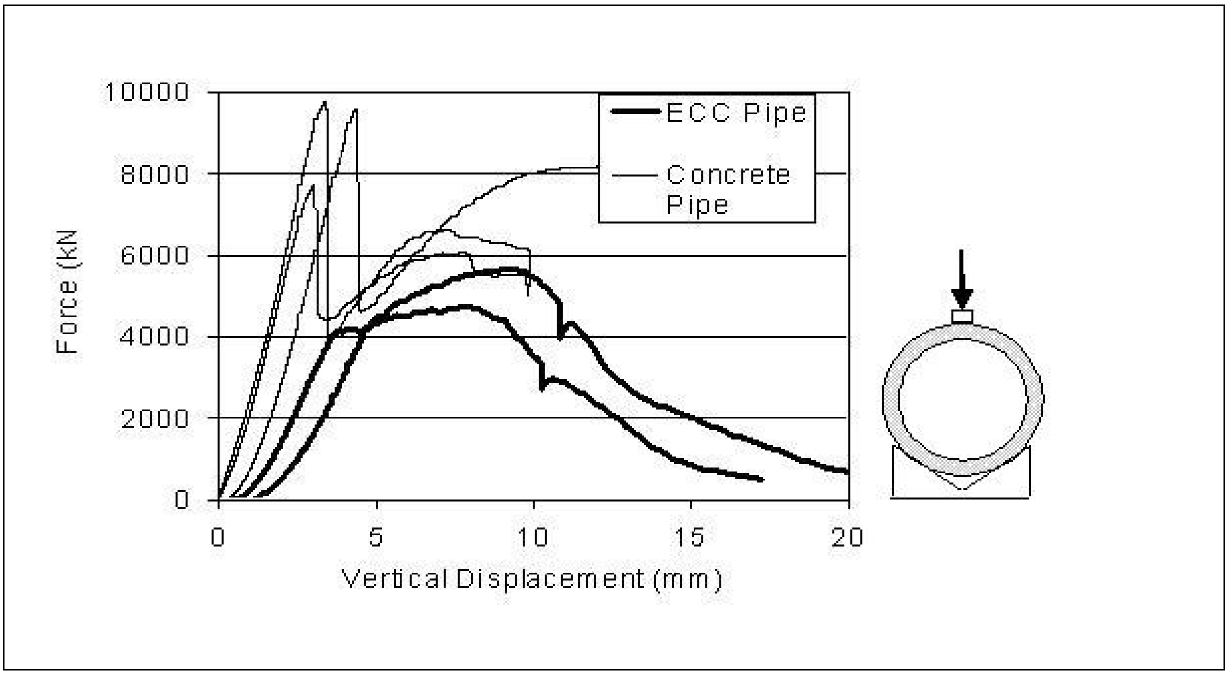


Figure 73: Pipe crushing results comparing fibre reinforced ECC pipe and steel-mesh reinforced concrete pipes, both manufactured with spinning technique

8. Fibre Matrix Interface Friction Bond Strength

The primary mode of failure, either fibre breakage or pullout from the matrix, influence the mechanical behaviour of the composite. The main advantage of fibre failure as a principle failure mode is that it generates high strength, but it causes relatively brittle behaviour. Fibre pullout as a principle failure mode on the other hand leads to increased toughness. The primary mode of failure is controlled by the fibre-matrix interface. If the critical bond-strength value is exceeded, the failure mode will be fracture instead of pullout. Therefore, the bond-strength controls the critical length, which is the minimum length, required to develop the full-strength capacity of the fibre.

In the case of PVA fibres, high interfacial frictional stress and a strong chemical bond with the surrounding cement-based phases due to its hydrophilic nature, can result in fibre surface damage during the pull-out process, as well as slip-hardening behaviour (refer Figure 74). Consequently, severe fibre rupture at failure is introduced in the composite.

It was postulated in section 6.6 that an increase in density is coupled with an increase in interfacial bond strength of the composite. With increased composite density, strength and matrix toughness of the fibre composite in the direction of the aligned fibres, the results in Figure 63 show the influence of the cast and piston extrusion manufacturing process on the density. The three point bending tests of the plate specimens show how the mechanical compaction of the extrusion process increased the interfacial bond strength to such an extent that, in collaboration with the improved fibre orientation, the tensile MOR increased by a factor of 2.7 (Figure 52).

In most cases where all the other parameters are the same, the lower the matrix toughness, the easier it is to achieve ductile behaviour of the composite. This phenomenon is contradicted with steel fibres. These fibres have such high tensile strength values, that it requires high matrix densification to alleviate insignificant fibre pull-out behaviour. One can postulate that a ‘balance’ needs to be obtained whereby the fibre tensile capacities are utilised and stretched to obtain overall higher composite tensile response.

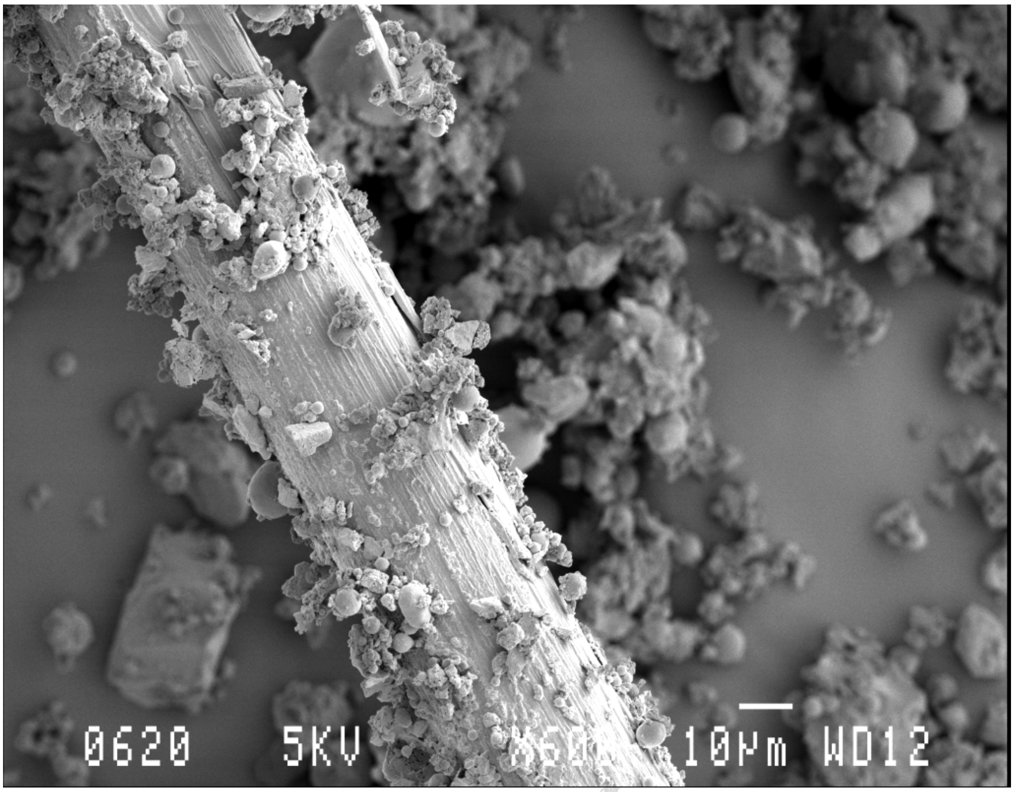


Figure 74: Fibre surface of piston extrudate (X600)

Fibre surface oiling treatment can alter interfacial properties, especially the chemical bond and the frictional stress.

Proper control of extrusion composites and design of the die are not only important for fibre orientation but contribute to the densification of the matrix and fibre packing to achieve a low porosity extrudate, as well as strengthen the interfacial bond between fibres and the matrix.

Another way of influencing the interfacial behaviour is through matrix particles packing around fibres. If well-rounded, fine particles in the matrix pack around fibres (Figure 74) the preferred pull-out mode of failure is activated (Figure 11). Extrusion assists in suitable packing of fines, proved by enhanced performance of extrudates with increased amounts of well-rounded fine particles. No such evidence yet exists for the fabrication processes casting and spinning.

9. Synthesis & Conclusions:

At the onset of a comprehensive research program to characterize the influence on ECC mechanical behaviour when subjected to extrusion, this research has reported the potential of beneficial increased alignment of fibres, accompanied by matrix–fibre interface strengthening through densification by the high pressure formation process. Thereby, the composite material mode of failure may be altered to cause rupture, in turn causing strong, but brittle composite tensile response. The inability of such brittle tensile response to exploit the post-first cracking plateau, or even strain hardening for stress redistribution to its full potential in loading cases causing stress gradients, was illustrated by flexural (three point bending) analysis.

Subsequently, a mixture of experience gained through preliminary experimentation on crude extrusion facilities of both piston and auger action types, as well as research results from the literature was presented towards identifying the major mechanisms at play in extrusion processing of ECC.

Based on the results of tests and literature study, it was concluded that:

<u>Conclusions drawn for primary and secondary research objectives:</u>

Development of equipment to manufacture composite components

1. The type of manufacturing process of ECC has a significant influence on the fibre orientation. Orientation of fibres is mainly in the load bearing direction of the process driving the mechanism. While piston extrusion aligns the fibres in the direction of extrusion, auger extrusion aligns the fibres diagonally. However, casting leaves the fibres orientated randomly. This leads to enhanced mechanical properties in the direction of main fibre orientation for the extruded composite.
2. The initial aim of developing the extrusion process evolved into an in-depth study of the effects of the different manufacturing process with regard to fibre orientation and interfacial bond strength. Due to time constraints, fibre pull-out and fibre orientation was not quantified. With regard to future development in this regard, kindly refer "Recommendations and Envisaged Research".

3. The primary objective as outlined in Chapter 1 was achieved: the effectiveness of the manufacturing process with respect to fibre orientation was substantiated.

Failure mechanisms of composite materials

4. Interfacial bonding between fibres and the matrix is coupled with the primary mode of failure. Depending on the interfacial bond, an optimal fibre length must be chosen. Since extrusion produces high compaction of ECC, reducing the porosity, the optimal fibre length for extruded composites is shorter than that for cast composites, in terms of improved mechanical performance.
5. Fibre breakage generates higher composite peak strength, whereas fibre pull-out leads to increased ductility.
6. Extrusion provides an increase in shear resistance between the fibres and the matrix (Shah et. al., 1997). If no fly ash is used in the mix, the shearing resistance of the fibre-matrix interface is increased to such an extent that fibre breakage may become the dominating failure mechanism. If fly ash is introduced in the extrusion mix, the lower matrix strength counteracts this mechanism, hence allowing fibre pull-out as the primary mode of failure, enabling favourable composite strain hardening behaviour. This was not shown in this research since we worked with steel fibres.

<u>Conclusions drawn on aspects that were not part of the aim of the investigation:</u>

Failure mechanisms of composite materials

7. The piston extruded pipe specimens exhibit anisotropic behaviour in the crushing and flexural tests due to the 'hinge-like' failure mechanism around each fibre. Because the fibres are aligned axially, the dominating mode of failure in a crushing test is axial cracking, across which virtually no fibres span.

Composite rheology

8. Extrusion is sensitive to different fibre lengths for the same fibre volume ratio. The viscosity of the matrix increases as the short fibre content increase.
9. Extrusion requires a dough-like mix. Clustering of fibres caused by low workability of the mix, or by too long fibres, reduces the dispersing of fibres in the matrix. This creates areas of weakness, allowing brittle failure.

Design of composite material mixes

10. Earlier research studies prove that durability of FRC and ECC is a function of the composite ductility and this phenomenon was evident in this research. More ductile composites generate more durability and serviceability, increasing the durability of structures.

Composite material properties

11. Piston extruded steel fibre reinforced pipes are more expensive than auger extruded polyethylene polyduct stormwater pipes, but they are cheaper than asbestos cement sewer pipes, uPVC water pipes and uPVC sewer pipes.

10. Recommendations and Envisaged Further Research:

Constituents:

1. Further research of different fibre types, aspect ratio and 'deformed' fibres with better mechanical bond could be conducted for the different manufacturing processes. This study would establish whether it is possible to increase the ductility and/ or the peak tensile strength.
2. The influence of fibre properties on fibre-matrix interfacial bond characteristics could be determined with the aid of a fibre pull-out apparatus (slip-controlled) test. This study should be coupled with the influence that the aligned fibres for the various processing techniques have on the composite strength characteristics before and after cracking.
3. Fibre properties have an influence on fibre distribution issues such as fibre orientation, segregation and clumping. Steel fibres for example exhibit good fibre orientation behaviour, which might not be the case with other 'less rigid' fibres such as PVA fibres. Whereas steel fibres segregate easily, other fibres (such as PVA) have the fibre clumping problem.
4. Brass coated steel fibres limit corrosion only to some extent. Rigorous mixing, as well the manufacturing technique, contributes to the corrosion of the steel fibre coating and instigates rusting of the steel fibres. A study could be conducted to investigate corrosion quantification, as well as corrosion reduction.
5. A possible study to quantify the influence of chemical additives, such as super-plasticizer and viscous agent, on the EECC strength, density

Extrusion technique:

6. Polymeric fibres show a lack of stiffness in the current mix rheology that influence desired fibre orientation negatively. Another aspect in the piston extrusion process is to introduce a draining section whereby the fresh material is consolidated in the transition or the die of the extruder. The fresh material in this case contains excess amounts of water that is squeezed out of the holes in the consolidation zone. The high water content could ensure better fibre distribution and orientation
7. The influence of the extrusion technique on matrix characteristics should be studied and quantified.

Testing/ Analysis:

8. Further investigation into the densification of FRC and ECC would provide a better understanding of the interfacial bond strength, as well as the overall composite strength and ductility generated. Suggestions on the quantification of the density cultivated by the various manufacturing techniques (casting, spinning, piston extrusion and auger extrusion) include:
   - pressure testing of manufactured pipes
   - permeability testing
   - density testing
9. The anisotropic behaviour of piston extruded products should be addressed. The processing effects of piston extrusion can't be changed, but the cross-sectional shape and size of the extrudate should be analysed and modelled to see which failure mechanism governs.
10. Cracks have the potential of induced corrosion of steel reinforcement, which in turn leads to decreased serviceability and service life of the structure. The possible crack size reduction or self-healing process of ECC's and FRC's must be looked at, and the rate of crack self-healing should be determined as to assist in the design process. This would for example set the agenda for the amount of prestressing needed to prevent excess cracking, and in doing so prevent maximum allowed corrosion before self-healing of cracks.
11. Statistical analysis of ECC and FRC cracking is yet another aspect that requires investigation. Statistical analysis of crack widths with the aid of image analysis technique can improve quantitive information on cracking. Crack formation characteristics are important for serviceability and durability.
12. The influence of higher ductility and energy absorption capacity on the behaviour of FRC under dynamic loading.
13. The strength and ductility of SFR-ECC must be increased. This could be achieved with hybrid fibre concretes (HFC). Actively interfering with the micro-crack process and macro-crack growth, HFC displays hardening behaviour by bridging the cracks and keeping the crack widths small. It is therefore suggested that a study is conducted on HFC composites, and study should be launched on the extrusion of HFC.

14. Coupled with the above-mentioned recommendation, HFC ensures proper fibre distribution. There is a contradictory trend in the optimal fibre length for the various processes. Extrusion requires shorter fibre lengths than that of cast composites. This is yet another reason why the introduction of HFC could have a positive influence on fibre distribution and fibre orientation.
15. Although the CT scans provided evidence of fibre orientation in the direction of the process driving the mechanism, quantification may be achieved by conductivity tests whereby a high conductivity measurement implicates uniform fibre dispersion and vice versa (Ozyurt et al., 2004).
16. To fully quantify fibre-matrix interfacial bond strength, fibre pull-out tests are needed whereby the tensile strength of a single embedded fibre is measured.
17. When pipe crushing tests are performed, they undergo cross-sectional ovalling under overload. Buried pipes will activate the horizontal earth pressure and thus have a substantial amount of residual load-bearing capacity at their disposal. From all this it will be apparent that the incorporation of steel fibres in the concrete has a by no means negligible effect in increasing the strength of pipes. Load bearing tests simulating buried and compacted pipes should be developed to ascertain the load-bearing capacity of pipes under earth loading, and how it compares to standard crushing as well as flexural tests.

Bibliography:

1. Akkaya, Y., Peled, A., and Shah, S.P., Parameters related to fibre length and processing in cement composites. *Mater. Struct.,* 33, (2000), 515-524.
2. Bentur, A. and Mindess, S. *Fibre Reinforced Cement-based Composites,* Elsevier Applied Science, Kent, UK. (1990).
3. Boshoff, W.P. & Van Zijl, G.P.A.G. 2003. Computational strategies for time-dependant behaviour of ECC, *Computational Modeling of Concrete Structures*, Bicanic et al. (eds) Swets & Zeitlinger, Lisse, 59-65.
4. Delvasto, S., Naaman, A.E., and Throne, J.L., Effect of pressure after casting on high strength fibre reinforced mortar, *Int.J. Cem. Compos. Lightweight Concr.*, 8(3), (1986) 181-190.
5. Igarashi, S., Bentur, A., and Mindess, S. The effect of processing on the bond and interfaces in steel fibre reinforced cement composites. *Cem. Concr. Compos*., 18, (1996) 313-322. In co-operation with: Jörg Dietrich, Thomas Berger, Bernd Feyerabend, Eberhard Geißler, Klaus Guthoff, Reinhold Koenning, Frank Tallarek.
6. Jordaan, C.R. and Van Zijl, G.P.A.G., Pipes of fibre reinforced sement, Final year (fourth year) B.Eng. thesis report, Thesis number S17, Stellenbosch, South Africa, November (2003).
7. Kearsley, E.P., Mostert, H.F., Optimizing mix composition for steel fibre reinforced concrete with ultra high flexural strength, The Sixth RILEM Symposium on Fibre-Reinforced Concretes (FRC) – BEFIB, 20-22 September (2004), 505-514.
8. Lee, C.D. and Soroushian, P., Distribution and Orientation of Fibres in Steel Fibre Reinforced Concrete, ACI Materials Journal, V.87, No.5, September-October (1990) 433-439.
9. Li, V.C., Mishra, D.K., Wu, H.C., Matrix design for pseudo-strain hardening fibre reinforced cementitious composites, Material and Structures, 28, (1995), 586-595.
10. Li, V.C., Takashima, H., Miyagai, K. and Hashida, T., A design approach for the mechanical properties of polypropylene discontinuous fibre reinforced cement-based composites by extrusion moulding, Engineering Fracture Mechanics 70 (2003) 853-870.
11. Li, V.C., Wang, S. and Wu, C., Tensile strain hardening behaviour of polyvinyl alcohol engineered cementitious composite (PVA-ECC), ACI Materials Journal, V.98, No.6, November-December (2001), 483-492.

12. Li, V.C., Wang, Y., Backer, S., A special technique for determining the critical length of fibre pull-out from a cement matrix, Journal of Materials Science Letters 7 (1988), 842-844.
13. Li, V.C., Maalej, M., Flexural/ Tensile-Strength ratio in Engineered Cementitious Composites, Journal of Materials in Civil Engineering, Vol.6, No. 4, November (1994) 513-528.
14. Maidl, B.R., *Steel Fibre Reinforced Concrete*, Ernst & Sohn, VCH publishing April (1996).
15. Narayanan, R. and Darwish, I.Y., Design chart for reinforced and pre-stressed fibre concrete elements, The Structural Engineer, V.68, No.2, 23 January (1990) 34-39.
16. Nilufer Ozyurt, Leta Y. Woo, Bin Mu, Surendra P. Shah, Thomas O. Mason 2004. Detection of fibre dispersion in fresh and hardened cement composites. Unpublished research paper from Centre for Advanced Cement-Based materials, North Western University, Evanston, USA.
17. Shah, S.P., Peled, A., Processing Effects in Cement-based Composites: Extrusion and Casting, Journal of Materials in Civil Engineering © ASCE, March/April, (2003) 192-199.
18. Shah, S.P., Akkaya, Y., Picka, J., Spatial distribution of aligned short fibres in cement composites, Journal of materials in civil engineering, August (2000), 272-279.
19. Shah, S.P., Aldea, C., Marikunte, S., Extruded Fibre Reinforced Cement Pressure Pipe, Advanced Cement Based Materials V. 8 (1998), 47-55.
20. Shah, S.P., Shao, Y., Marikunte, S., Extruded fibre-reinforced composites, Concrete International, April (1995), 48-51.
21. Shah, S.P., Shao, Y., Mechanical properties of PVA fibre reinforced cement composites fabricated by extrusion processing, ACI Material Journal, V. 94, No.6, November-December (1997), 555-564.
22. Shah, S.P., Srinivasan, R., DeFord, D., The use of extrusion rheometry in the development of extruded fibre-reinforced cement composites, Concrete Science and Engineering, Vol.1, March (1999), 26-36.
23. Song, G. and Van Zijl, G.P.A.G., Tailoring ECC for Commercial Application, The Sixth RILEM Symposium on Fibre-Reinforced Concretes (FRC) – BEFIB, 20-22 September (2004), 1391-1400.
24. Stang, H., Extrusion of ECC-material, 1-10 [1999].

25. Van Mier, J.G.M., Nakamura, S., Masuda, Y., Self compactibility of hybrid fiber concrete containing PVA fibers, The Sixth RILEM Symposium on Fibre-Reinforced Concretes (FRC) – BEFIB, 20-22 September (2004), 525-535.
26. Yamada, K., Ishiyama, S., Determination of fiber contribution in composites employing tension softening properties, The Sixth RILEM Symposium on Fibre-Reinforced Concretes (FRC) – BEFIB, 20-22 September (2004), 443-452.

<u>Standards and Codes of Practice:</u>

27. SABS 819:2001. Edition 3.1, Fibre-cement pipes, couplings and fittings for sewerage, drainage and low-pressure irrigation.
28. SANS 5863/ SABS SM 863:1994, Concrete tests – Compressive strength of hardened concrete.
29. SANS 5864/ SABS SM 864:1994, Concrete tests – Flexural strength of hardened concrete.
30. CI Manual of Concrete Practice Part 6 – 2003
    Design considerations for steel fibre reinforced concrete
31. ASTM sand grading manual
    U.S. Silica's ISO 9002 Sertified Quality System
32. Spider 8 PC-MEsselektronik User Manual. Hottinger Bladwin MEsstechnik GmbH, Darmstadt, Germany.
33. Zwick Z250 S5NA Materials Testing Manual. Instruction Manual, Material Testing Machine Data, Zwick GmbH, Ulm, Germany.

Appendix:

1. Cost (and Viability) Analysis:

Mix design impacts the manufacturing cost of the composite and assessing whether the optimum or chosen mix design for a specific process is viable. However, the intent of this research was to develop extrusion processes for ECC. Subsequent research and development is required for commercialising this processing concept.

The practical application of ECC extrusion to manufacture pipes possibly lies in the use for smaller storm water pipes, effluent or even water reticulation.

The existing products on the market are:

A) Polyduct stormwater pipes are manufactured specifically for storm water reticulation. This is manufactured with the auger extrusion method. The constituents mainly comprise of polyethylene that gets melted into the auger extruder (similar to uPVC pipes), but the pipes comprise of an inner and outer section that gets extruded all in one process. Listed below is the cost (priced for a purchase of 300m pipe length and excludes VAT):
   160mm diameter polyduct stormwater pipe ... > R30-00/m

B) uPVC pipes are manufactured for water- and sewer reticulation. It is manufactured by auger injection moulding. Listed below are the costs (priced for a purchase of 300m pipe length and excludes VAT):
   160mm diameter CL.12 uPVC pipe ... > R98-00/m
   160mm diameter CL.9 uPVC pipe ... > R76-69/m
   200mm diameter CL.12 uPVC pipe ... > R150-83/m
   200mm diameter CL.9 uPVC pipe ... > R120-00/m
   315mm diameter CL.34 heavy duty sewer pipe…>R235-00/m

C) Alternatively, asbestos sewer pipes could be used. Listed below is the cost (priced for a purchase of 300m pipe length and excludes VAT):
   300 AC sewer pipe (including couplings) …>R182-00/m

If we now consider piston extruded pipes with an OD=180mm and an ID=150mm, we get a volume of 0,005m^3/m length of pipe. If we consider a typical extrusion mix, for example the mix in section 4.1.1, we get a cost of R6200/m^3. This works out to R 48-20/m. This is very expensive, mainly because of the high steel fibre volume (42% of the total mix price) and the high viscous agent percentage. Viscous agent is very expensive (approximately R180-00/kg), which then accounts for 36% of the total mix price. If we add some manufacturing costs to the R 48-20/m, we should get something in excess of R55-00/m.
However, if we reduce the viscous agent from 0,8% to 0,5% and the steel fibre volume from 3% down to 2%, the total mix price gets reduced by 32%. This yields a price estimate of R 40-00/m.

Important:
This price excludes the price of couplings or concrete sealing for pipe connections. The piston extruded pipes are more expensive than the polyduct pipes. Prices listed are SA market Rand prices as at October 2004.
The supplier for the prices of manufactured products as listed above was C.I. Merchandising (Pty) Ltd.