The electro-mechanical tensile properties of an engineered cementitious composite

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Manuscript title: The electro-mechanical tensile properties of an engineered cementitious composite

Authors: D. Saraireh, B. Suryanto, W. J. McCarter and S. Walls

Affiliation: School of Energy, Geoscience, Infrastructure and Society, Institute for Infrastructure and Environment, Heriot-Watt University, Edinburgh, EH14 4AS, Scotland, UK

Corresponding author: B. Suryanto, School of Energy, Geoscience, Infrastructure and Society, Institute for Infrastructure and Environment, Heriot-Watt University, Edinburgh, EH14 4AS, Scotland, UK. Tel.: +44-131-451-3817; Fax: +44-131-451-4617.

E-mail: b.suryanto@hw.ac.uk
Abstract

The influence of ongoing cement hydration and multiple micro-crack formation on the electrical impedance of an engineered cementitious composite (ECC) is presented. Impedance measurements are obtained over the frequency range 20Hz-1MHz and displayed in Nyquist format; in addition, the permittivity and conductivity were de-embedded from the measured impedance and presented in both the time and frequency domains to elucidate the nature of conduction and polarization processes. It was shown that over the 90 d curing period, the ECC displays a classic impedance response. Both conductivity and relative permittivity are shown to be frequency dependent due to bulk relaxation processes operative within the composite. Tensile straining was shown to result in a detectable change in the impedance response, but retains a similar overall profile. When presented in the frequency domain, a downward displacement in both conductivity and relative permittivity profiles was evident with increasing tensile strain. It is shown that the relative permittivity at the high-frequency end could be exploited as a potentially useful indicator for strain/damage detection. The influence of micro-cracking on the piezo-resistive response of the composite is discussed based on crack patterns obtained from both visual observations and digital image correlation.

Keywords: Electrical properties; composite materials; characterisation techniques; fibre-reinforcement; non-destructive testing
Introduction

Concrete is the most widely used construction material, with a large proportion of infrastructure worldwide built using concrete in one form or another. Whilst most key infrastructure is designed for an intended working life of at least 100 years, premature deterioration remains commonplace (Li et al., 2009; Kim et al., 2016). Although some deterioration may not have a direct implication on structural integrity, immediate repair may be required so as not to impair in-service performance (Frangopol et al., 2017). The earlier any deterioration is detected, the earlier appropriate remedial measures can be taken thereby avoiding the need for costly repairs at a later stage (Pines and Aktan, 2002; McCarter et al., 2017). With the ever-increasing amount of infrastructure around the world, and the continual deterioration of existing infrastructure, it is expected that structural inspection and maintenance will have an increasing role to play for the foreseeable future.

It is common practice to undertake periodic visual inspections to enable effective management and maintenance of infrastructure (Pines and Aktan, 2002; Omar and Nehdi, 2018); however, this requires direct site visits by qualified engineers at predetermined times during the life of a structure (Highways England, 2017). Whilst visual inspection is relatively straightforward to undertake and allows evidence to be gathered directly on-site, it only provides a snapshot of information in time; it is the change in condition with time which needs to be assessed so as to provide a reliable assessment of structural health (Choi et al., 2004; Hedegaard et al., 2017; Kim et al., 2018). A further limitation of visual inspection is that some damage may not be readily visible, thereby remaining undetected until advanced stages which are costly to repair. A number of automated, structural health monitoring systems have been developed to mitigate the shortcomings of traditional on-site inspections, with some already implemented in real structures and bridges (Agdas et al., 2015; Abé and
monitoring systems typically require the installation of an extensive network of surface mounted or embedded sensors and monitoring of their response over time. A similar approach is employed in the work presented, although the concrete itself is used as the sensor. Developments in the field include moisture sensors (McCarter et al., 2012), weight in-motion and traffic sensors (Shi and Chung, 1999; Han et al., 2011) and strain/damage sensors (Azhari and Banthia, 2012; Yoo et al., 2019). Whilst the sensing capabilities of cement-based strain/damage sensors have been well documented, they may only be able to be employed under limited damage levels as the relationship between damage and strain increases nonlinearly after cracking.

Recently, a damage-tolerant cement-based material known as the Engineered Cementitious Composite (ECC) has been identified as a potential damage sensor (Hou, 2008; Hou and Lynch, 2005; Lin et al., 2011; Ranade et al., 2014). The damage-tolerant property of ECC originates primarily from its ability to exhibit multiple-fine cracking when subjected to tensile stresses beyond the elastic range, generally displaying tensile strain hardening behaviour with a tensile capacity in the order of a few percent and an average crack width under 100µm at maximum strain (Li, 2008; Suryanto et al., 2015; Lee et al., 2018; Chen et al., 2018; Frank et al., 2018; Ma et al., 2019). This differs from ordinary concrete and fibre reinforced concrete which, generally, fail locally by a single crack. With regard to damage sensing, work has predominantly focussed on the piezo-resistive properties of this composite. Hou (2008) employed the four-point measurement technique to study the piezo-resistivity of the standard ECC mix under both d.c. and a.c. currents, with the latter applied at 5kHz. They demonstrated that the composite displays a quasi linear piezo-resistive response during strain-hardening resulting from the formation of multiple micro-cracks. Hou and Lynch (2005)
further studied the piezo-resistive response of ECC mixes containing steel and carbon fibre using the four-point d.c. measurement. They demonstrated that the ECCs display a linear relationship between the change in resistivity and tensile strain, and found that these ECCs were less sensitive to mechanical strain due to the presence of crack-bridging conductive fibres. To minimise bridging effects, Lin and co-workers (2011) added carbon black to the standard ECC mix to increase the contrast of the composite resistivity before and after cracking. The four-point a.c. measurement technique was employed, with measurements undertaken at 100Hz. It was found that the sensitivity of the composite to cracking improved. Employing the two-point method and a constant a.c. current of 1kHz, Ranade and co-workers (2014) tested moderate- and high-volume fly-ash ECC mixes with different micro-crack characteristics. It was found that, although the high-volume fly-ash mix exhibited a greater number of micro-cracks, it exhibited less sensitivity to cracking than the moderate-volume fly-ash mix due to its smaller micro-crack widths.

To date much of the work has studied the piezo-resistive properties of this composite using d.c. and fixed-frequency, a.c. resistivity measurements and little attention has been directed towards investigating the electrical properties of ECC over a wide frequency spectrum. When subjected to an alternating current, porous materials such as the ECC can be expected to display both conductive and capacitive behaviour: the former due primarily to ionic conduction processes and the latter due to polarization processes operative within the material (Suryanto et al., 2016; Suryanto et al., 2018a). Measurement of both quantities could provide more detailed information with regard to strain/damage sensing capabilities of ECCs. In addition to mechanical loading, it is anticipated that the electrical properties of the composite are also influenced by cement hydration, but this aspect has been overlooked in prior studies. These aspects are addressed in this work.
Experimental Programme

Materials and mix proportions

The mix proportions for the ECC used in this experimental programme are presented in Table 1. The binder comprised a blend of CEM I 52.5N Portland cement to BS EN197-1 (BSI, 2001) and fine fly-ash (Superpozz SV80, supplied by ScotAsh Ltd) at a cement/fly-ash ratio of 1:1.8. To improve the dimensional stability of the mix, fine silica sand with a mean particle size of 120µm was added at a sand/cement (S/C) ratio 0.6:1 by mass; this particle size and amount were considered to ensure that the matrix toughness is low enough to allow tensile strain hardening behaviour. A typical oxide analysis of these materials is presented in Table 2. The water/binder (w/b) ratio was set at a relatively low value (=0.28) which produced a mix with adequate viscosity for fibre dispersion. The only fibres incorporated into the mix were Kuralon K-II REC15 polyvinyl alcohol (PVA) fibres, which have been specifically developed for use in ECC by Kuraray Japan. The fibres had an average length of 12mm, diameter of 39µm, tensile strength of 1.60GPa, and were supplied with a proprietary oil-based coating agent (1.2% by mass) to reduce excessive bonding with hardened ECC matrix. To aid fibre dispersion and improve the workability of the mix, high-range water-reducing (HRWR) admixture (MasterGlenium ACE499) was added at a dosage of 1% by cement weight.
Test specimens, fabrication and curing

A 10-litre Hobart planetary motion mixer was used to prepare the mix presented in Table 1 in a single batch. In total, thirteen specimens were produced: three 40×40×160 mm (long) prismatic specimens (P1-P3) to monitor the influence of continued hydration, four dog-bone shaped specimens (DB1-DB4) of dimensions in accordance with the JSCE recommendation (JSCE, 2008) to determine the mechanical and electrical properties under uniaxial tensile loading, and six 50mm cuboidal specimens (C1-C6) to determine compressive strengths.

The prisms had two 45×65×2 mm (thick) perforated stainless steel electrodes placed 140mm apart (see Figure 1a). The electrodes had 10mm round holes at a 15mm staggered pitch to allow ease infiltration by the ECC during casting and thereby ensuring intimate bonding (Suryanto et al., 2018b). The dimensions of the dog-bone shaped specimens are presented in Figure 1b which also displays the electrode configuration employed, comprising two wire electrodes wrapped securely at the opposing ends of the central neck of the specimens. The wire electrodes were covered with several coats of silver-loaded conductive paint to seal any gaps and ensure intimate contact with the specimen.

The prisms were cast into a single-use 3-gang polystyrene mould, whereas the dog bone and cube specimens were cast in a custom-made Plexiglas mould and 3-gang steel moulds, both of which had been treated with a proprietary release agent (Sika Everbuild 206). Immediately after casting, all specimens were covered with thick cling film and allowed to cure for 24 hours. The specimens were then demoulded and stored in a curing tank at 20±2°C until they were required for testing. After two weeks, the dog-bone specimens were removed from the curing tank and allowed to dry for ~3 hours; the wire electrodes, discussed above, were then attached to the specimens and coated with the conductive paint. When the paint was dry, the samples were returned to the tank until required for testing at 28 days.
Test procedures and equipment

The work undertaken involved the testing of three prisms to study the influence of on-going cement hydration and the testing of four dog-bone shaped specimens to obtain the electro-mechanical properties of the composite under tensile straining.

Hydration study and compressive strength

A Keysight E4980AL precision LCR meter was employed to acquire the electrical impedance of prisms P1–P3 over the initial 90 days after casting. The impedance was recorded at 20 spot frequencies per decade over the frequency range 20Hz–1MHz. The LCR meter was operated in voltage drive mode, at a constant signal amplitude of 350mV, and controlled by a desktop computer (PC) which was also used for data acquisition. Communication with the LCR meter was established via the built-in USB interface in the LCR, which was accessed by the PC through a Keysight IO Library Suite software (Version 2017.1). To manage the overall running of the experiment, a customised virtual instrument was developed in the LabVIEW environment (LabVIEW, 2017), which permits a full control of the LCR meter. This virtual instrument was also used to process the raw impedance data, present the results in both tabular and graphical forms, and store the processed data in a CSV format for further analysis. In the electrical measurements, four individually screened, short coaxial leads were used. At the time of testing, leads from the high current (H\text{CUR}) and potential (H\text{POT}) terminals on the LCR meter were connected to one electrode and the low current (L\text{CUR}) and potential (L\text{POT}) to the other, following the two-point configuration (McCarter \textit{et al.}, 2015).

In addition to electrical measurements, the compressive strengths of the ECC were determined from cubes C1–C6 using a 3000kN Avery–Dennison testing machine, under a loading rate of 50kN/min. The strengths obtained on the 28\textsuperscript{th} and 90\textsuperscript{th} days of curing denoted, respectively, $F_{28}$ and $F_{90}$, are presented in Table 3, together with the tensile strain capacity $\varepsilon_{tu}$. 
first crack strength $f_{cr}$, and ultimate tensile strength $f_{tu}$ determined from the dog-bone specimens (DB1–DB4).

Piezo-impedance study

On the 28th day of curing, tensile testing was performed on the dog-bone shaped specimens (DB1–DB4) using a 100kN 4206 Instron (see Figure 2a). Prior to testing, each specimen was aligned in the machine and clamped at both ends using pneumatic grips. Loading was then performed under a crosshead speed of 0.5mm/min. Tensile stresses were determined by dividing load cell readings from the test machine by the cross-sectional area of the narrower (central) section (approximately 30x12mm$^2$), whereas the longitudinal strains within this region were determined from the average of two linear variable differential transducers (LVDTs) readings. These LVDTs were attached prior to testing at the bottom end of the bone-neck region, one on each side, through two lightweight plastic mounting blocks (see Figure 2b). The gauge length was ~60mm. All data were recorded using a 16-bit USB data acquisition system at a sampling rate of 1 second.

In addition to the stress and strain readings, simultaneous electrical measurements were undertaken during the loading process to study the influence of multiple micro-cracking, using the same measurement system employed for testing the prisms. Prior to testing, the pair wire electrodes at the opposing ends of the central neck of each sample (the distance was ~80mm) were connected to the LCR meter through individually screened coaxial leads, with the connection at the electrodes using alligator clips (see Figure 2a). Electrical measurements were then undertaken throughout the installation process and during tensile loading at thirteen spot frequencies covering five decades: 100Hz, 200Hz, 500Hz, 1kHz, 2kHz, 5kHz, 10kHz, 20kHz, 50kHz, 100kHz, 200kHz, 500kHz and 1MHz. The low frequencies (<100Hz) were omitted from the sweep to minimise the duration of the measurement. This sweep
measurement was repeated over a 3-sec cycle, facilitating virtually continuous, real-time monitoring during loading thus minimizing the influence of time-dependent effects on the measured impedance.

To provide evidence of micro-crack formation, digital images of the front face of the dog-bone specimens were taken at 0.1mm displacement increment using an 18.4MP Nikon 1 J4 mirrorless digital camera, positioned at approximately 300mm from the specimen. To remove inadvertent camera movement, images were collected remotely using the Nikon wireless mobility utility smartphone application (Version 1.2.1). Prior to testing, random black dots were manually drawn on the front surface of the dog-bone specimens which had been given a thin coat of white acrylic paint (see Figure 2b). This was undertaken to give a random pattern with a sharp contrast, thereby facilitating automated strain mapping within the boundary indicated in Figure 2b. The images were then processed using the digital image correlation (DIC) freeware Ncorr (Version 1.2.1) (Ncoor, 2018; Blaber et al., 2015; Suryanto et al., 2017a; Tambusay et al., 2020). In addition to this automated crack mapping, more detailed crack mappings were undertaken manually using the ImageJ1 software (Schneider et al., 2012), with the aim at obtaining the number and width of each individual micro-crack under various stages of loading.

Data analysis and presentation

The impedance of a cement-based system, \( Z(\omega) \) in ohm (\( \Omega \)), subjected to a small-signal sinusoidal electric field at an angular frequency, \( \omega \), can be represented in a rectangular form as (McCarter and Brousseau, 1990; Starrs and McCarter, 1998; McCarter et al., 2002),

\[
Z(\omega) = Z'(\omega) - iZ''(\omega) \tag{1}
\]

where \( Z'(\omega) \) is the resistive (real) component and \( Z''(\omega) \) is the reactive (imaginary) component. These two parameters are commonly presented in the Nyquist format, with
Z''(ω) plotted against Z'(ω) over a wide frequency range (see Figure 3a). At any frequency, the impedance of the cementitious system will result from two superposed phenomena: conduction and polarisation which are quantified, respectively, by the bulk conductivity, σ(ω), and relative permittivity, κ'ε(ω), of the system. Both parameters are generally presented in the frequency domain (see, for example, the schematic presented in Figures 3b, c) to investigate the nature of conduction and relaxation processes. These parameters can be de-embedded from the resistive and reactive components through the relationships (McCarter et al., 2004; Suryanto et al., 2016),

\[
\sigma(\omega) = \left(\frac{Z'(\omega)}{Z'(\omega)^2 + Z''(\omega)^2}\right) g_p \tag{2}
\]

\[
κ'_ε(\omega) = \frac{1}{κ_0ω} \left(\frac{Z''(\omega)}{Z'(\omega)^2 + Z''(\omega)^2}\right) g_p \tag{3}
\]

with κ₀ representing the permittivity of free space (8.854×10⁻¹² Farads/m) and g_p representing the geometrical constant which is dependent upon the electrode geometry and spatial positioning of the electrodes within the system.

The value of g_p was obtained from a-priori experiments using solutions of known conductivity, using the same polystyrene mould and perforated stainless-steel electrodes as in the test prisms. A value of g_p = 88.48/m was obtained. For the dog-bone specimens, the calibration was done by converting the resistance measured from prisms P1–P3 at 28-days of curing, denoted R_p,28, to resistivity, ρ,28, through the relationship,

\[
ρ_{28} = R_{p,28} \frac{1}{g_p} \text{ Ohm-m} \tag{4}
\]

Accordingly, the geometrical constant for the dog-bone specimen, g_{db}, could be calculated,

\[
g_{db} = \frac{R_{db,28}}{ρ_{28}} /m \tag{5}
\]
where $R_{db,28}$ is the measured resistance for the dog-bone specimen after 28-days curing. $g_{db}$ was obtained as 245.28/m.

**Results and discussion**

Hydration study

Complex impedance response (no tensile loading)

The impedance spectra for prisms P1–P3 over the initial 90-day period are presented in Nyquist format in Figure 4a, with frequency increasing from right-to-left across the curve. For reasons of clarity, only every 5th data marker is highlighted on each curve. The solid lines represent the impedance spectrum of prism P1, while the dashed lines represent the spectra of prisms P2 and P3. It is evident from this Figure that there is excellent agreement between the notionally identical prisms with each response comprising three distinct features,

(i) A small spur at the low-frequency (right-hand) side of the spectrum;

(ii) A narrow *U-shaped* intermediate valley region; and

(iii) An arc at the high-frequency (left-hand) side which, with the intermediate plateau noted in (ii), represents the bulk impedance response.

The low-frequency spur represents the response resulting from polarization processes at the electrode/specimen interface (McCarter *et al.*, 1988; McCarter and Brousseau, 1990) and constitutes part of a larger arc that would only form at frequencies significantly lower than the lower frequency limit used in the present study, viz $\ll 20$Hz. The *U-shaped* intermediate region can be attributed to the presence of unburnt carbon in the fly-ash, the extent of which has been found to have direct proportion to the amount of free carbon content in the fly-ash (McCarter *et al.*, 2004; Suryanto *et al.*, 2017b) which is quantified by its loss-on-ignition (LOI) (see Table 2). By presenting the impedance data in Nyquist format, as in Figure 4a, it is possible to separate the response of the electrodes from that of the bulk material thereby
allowing calculation of the bulk ionic resistance of the composite. This was obtained from the projected intercept of the low-frequency end of the bulk arc with the real axis. By doing so, the bulk resistance at 7 days was evaluated as \( \sim 1.1k\Omega \), increasing to \( \sim 2.9k\Omega \) at 28 days, \( \sim 5.1k\Omega \) at 56 days and \( \sim 7.3k\Omega \) at 90 days. The progressive increase in bulk resistance is indicative of continual refinement in pore structure resulting from on-going cement hydration and pozzolanic reaction, causing a reduction in ionic conduction with time.

With reference to Figure 4a, it is evident that the progressive increase in bulk resistance is accompanied by displacement of the impedance spectrum to the right, implying an increase in the sample impedance over the entire frequency range. It is also interesting to observe that the definition of the bulk arc becomes more discernible with time, when measured within the same frequency range, viz 20Hz–1MHz. This manifests as a reduction in the cusp-point frequency with increasing curing time across the entire impedance spectrum. For illustrative purposes, Figure 4b displays the change in cusp-point frequency, \( f_c \), (i.e. the frequency corresponding to the minimum point within the U-shaped valley region) over the 90-day curing period, with the error bars representing \( \pm \) one standard deviation. A reduction in frequency is evident over the initial 21-days, followed by a more gradual decrease in frequency over the remainder of the test period reflecting on-going hydration and resulting microstructural changes within the composite. To provide an improved understanding of these mechanisms, the frequency dependent parameters (relative permittivity, \( \kappa'_c(\omega) \), and conductivity, \( \sigma(\omega) \)) are presented and discussed below.
Relative permittivity and bulk conductivity (no tensile loading)

In a heterogeneous system such as the ECC, a number of polarization processes can operate simultaneously within the system within an overlapping frequency range. Each process may relax according to its own time constant, which makes it difficult to accurately determine the contribution of each individual mechanism to the polarizability of the system.

The relative permittivity provides a relative measure of the polarizability of the system and, at any particular frequency of applied electrical field, quantifies the sum of all polarization mechanisms operative at that frequency. Presenting the permittivity in the frequency domain can, therefore, give a clearer view of the dominant polarization mechanisms (and the frequency range over which they operate). The relative permittivity, \( \kappa'_r(\omega) \), which has been de-embedded from the impedance data of prisms P1–P3 using Eq. 3, is presented in the frequency domain in Figure 4c. It is apparent from this figure that a region of dispersion exists, as evidenced by the progressive reduction in permittivity up to the upper frequency limit. There is also a distinct change in the rate of dispersion across the entire frequency range presented. Consider, for example, the permittivity at 7 days which has attained a value of \( \sim 3.4 \times 10^6 \) at 20Hz, this decreases by almost two orders of magnitude to \( \sim 2.2 \times 10^4 \) at 1kHz, \( \sim 370 \) at 100kHz, and to \( \sim 115 \) at 1MHz. Within the frequency range, it is postulated that three dominant mechanisms of polarization are operative:

(i) Polarization processes at the electrode/sample interface, referred commonly to as the electrode polarization (McCarter et al., 2009; Ishai et al., 2013; McCarter et al., 2015) and can result in anomalously high permittivity values. This is generally a low-frequency mechanism, typically relaxing at frequencies <kHz;
(ii) Double-layer polarization occurring due to charges electrostatically held on the surfaces of the cement gel and other particles such as fly-ash and fine sand (Schwan et al., 1962; Schwarz, 1962; McCarter et al., 2002). This is a low/medium frequency mechanism and may operate up to ~100kHz region; and

(iii) Interfacial or space charge polarization resulting from the accumulation of charges at the interface of two dissimilar materials with different relaxation times, also known as the Maxwell-Wagner polarization (Hasted, 1973; McCarter et al., 2002; Prodromakis and Papavassiliou, 2009; Iwamoto, 2012). In a cement-based system including ECCs, this can arise from translating charges which are blocked inside isolated pores and accumulate at pore water/hydrate interfaces. Interfacial polarization is an intermediate-frequency mechanism, operating typically over high kHz into the MHz region.

With reference to Figure 4c, it is proposed that the mechanism responsible for the decrease in permittivity at frequencies <1kHz is the relaxation of polarization processes at the electrode/sample interface, possibly overlapping with relaxation of double-layer process within the composite itself. Within the frequency range 1kHz-1MHz, it is proposed that the continuing decrease in permittivity is as a direct result of two superimposed mechanisms: the relaxation of double-layer processes, operating primarily within this frequency range, and the relaxation of interfacial (Maxwell-Wagner) processes, which have a more dominant influence at frequencies >100kHz.

It is also interesting to note from the results presented in Figure 4c that there is a reduction in the magnitude of the dispersion/relaxation with increasing curing time, from ~5 orders of magnitude at 7 days to ~3 orders of magnitude at 90 days. This is attributed, in part, to the reduction in relaxation time discussed above and to the change in the dispersive polarization
processes with time. To further investigate the dispersive behaviour of the material, Figure 4d presents the bulk conductivity in the frequency domain, with the conductivity de-embedded from the impedance measured from prisms P1–P3 using Eq. 2. Conductivity gives a measure of both ionic conduction through the movement of ions within the continuous capillary pore network, which is low-frequency feature, and a dispersive conduction contribution resulting from the relaxation of polarization processes which is dependent upon the frequency of the applied field. It can be inferred from the conductivity plots presented in Figure 4d that dispersion results in a very gradual increase in conductivity across the entire frequency range, indicating the existence of relaxation of polarization processes over the entire range. This would indicate that the polarization mechanisms operate at an overlapping frequency range resulting in a spread of relaxation time. Owing to the gradual increase, it would be difficult to separate the underlying mechanisms from this presentation formalism, with the exception of the conductivity at 56- and 90-day of curing in which a more noticeable increase in conductivity is evident at frequencies greater than ~100kHz. This is attributed primarily to the relaxation of interfacial processes, discussed above.

Piezo-impedance study

Complex impedance response under tensile loading

The complex impedance plots for specimens DB1 to DB4 during tensile loading conducted on the 28th day of curing are presented in Figures 5a–d. Owing to the number of data points in each curve (i.e. measurements at 13 spot frequencies), the data markers have been connected with B-Splines and, for reasons of clarity, only the response at 1% strain increment is presented. At any stage of loading, cracked ECC displayed a classic response comprising an arc forming the left-hand-side of the plot and a spur forming the right-hand side, which is not dissimilar to the response observed in the hydration study (see Figure 4a). It is apparent from
Figures 5a–d that tensile straining resulted in the entire response being gradually displaced to the right, indicating an overall increase in impedance with increasing strain. With reference to Figures 6a, b, this gradual increase is as a direct result of successive formation and widening of multiple micro-cracks (Saraireh et al., 2017; Wansom and Kanokkanchana, 2017), which have the effect of altering the conduction pathway within the composite, from a relatively straight (Figure 6a) to a more tortuous pathway (Figure 6b). Accordingly, as the tensile strain increases, this has the effect of reducing the continuity and increasing the circuitous path length, and hence increasing the overall impedance of the ECC. Regarding Figure 6b, conduction will occur via several possible mechanisms across the micro-crack as depicted in the figure; at this stage, however, it is difficult to delineate the contribution from each pathway.

Another interesting feature which is apparent from Figures 5a–d relates to the definition of the high-frequency arc associated with the bulk response of the material including the micro-cracks. It is observed that the arc becomes more pronounced as the strain increases, indicating a shift in time constant to the frequency range under investigation as in the hydration study.

Relative permittivity and bulk conductivity under tensile loading

Figures 7a–d present the relative permittivity computed using Eq. 3, plotted in the frequency domain with varying tensile strains and marked at every 1% increment of strain. In general terms, the trend in the relative permittivity plots is, in many respects, similar to the respective specimens in the hydration study (see Figure 4c), characterised by a reduction in relative permittivity with increasing frequency across the entire frequency range. As before, this is due to the relaxation of superimposed polarization mechanisms operative within the composite and the shift in time constant with increasing strain, which has the effect of shifting the relative permittivity curve to the left along the Z’ axis toward the origin.
It is interesting to note that whilst the relative permittivity plots presented in Figure 4c all merge at frequencies >~200kHz, this is not the case for the plots presented in Figures 7a–d which display a progressive downward displacement with increasing strain across the entire frequency range, including high frequencies (i.e. > 200kHz). This can be associated with the progressive formation micro-cracks within the ECC matrix; as the relative permittivity of the air-gap between two opposite cracked surfaces is ~1, multiple crack formation would have the effect of decreasing the overall polarizability of the system. To understand this aspect, consider the idealised system presented in Figure 8a comprising the saturated ECC matrix, and a crack comprising an air-gap and a crack-bridging pathway. The bridging pathway would represent all the possible pathways presented in Figure 6b, so could be regarded as a smeared contribution. Given that the micro-cracks are, in essence, connected in series with the bulk ECC matrix, the permittivity of the micro-cracks could be estimated by the mathematical mixing law (Reynolds and Hough, 1957),

\[
\frac{\phi_{r,t}}{\kappa_{r,t}} = \frac{\phi_{cr}}{\kappa_{cr}} + \frac{\phi_{r,i}}{\kappa_{r,i}}
\]

with \(\kappa_{r,t}\) representing the bulk relative permittivity of the composite at strain \(\varepsilon_t\) after the start of the test, \(\kappa_{cr}\) representing the average relative permittivity of the micro-cracks, and \(\kappa_{r,i}\) is the bulk relative permittivity of the composite at the start of the test (i.e. \(\varepsilon_t = 0\)). Both \(\kappa_{r,t}\) and \(\kappa_{r,i}\) were evaluated at the upper frequency limit which, in the present study, is 1MHz. With reference to the system displayed in Figure 8a, the total volume at strain \(\varepsilon_t\) after the start of the test is given by \(\phi_{r,t} = \phi_{r,i} + \phi_{cr}\), where \(\phi_{r,i}\) is the initial volume at the start of the test and \(\phi_{cr}\) is volume fraction of the micro-cracks at strain, \(\varepsilon_t\).
Figure 8b presents the average permittivity of the micro-cracks computed using Eq. 6 at discrete tensile strain levels. The results show that the value of the apparent permittivity of the micro-cracks is consistently higher than that of air (≈1), indicating the presence of other materials within the space between two micro-crack surfaces. The plots also display variations in calculated permittivity values, with a general decreasing trend with increasing tensile strain up to ~4% where the permittivity plateaus at ~4. The best-fit power equation was added to provide an alternative means of predicting the tensile strain in a fully saturated system from permittivity measurements. The variation in permittivity would indicate the spatial distribution in the number of physical pathways across the crack (see Figure 6b), whilst the reduction in permittivity with increasing strain is indicative of their temporal change during the loading process.

To further highlight the influence of crack formation on the bulk electrical properties of cracked ECC, the bulk conductivity of specimens DB1-DB4 at varying strains is presented in the frequency domain in Figures 9a–d. As before, owing to the relaxation of the polarisation mechanisms operative within the ECC matrix, the conductivity gradually increases with frequency across the entire frequency range. Tensile straining is shown to result in a downward, parallel displacement of the bulk conductivity curves, resembling the trend displayed in Figure 4d. This is as a direct result of the formation of micro-cracks with increasing load rather than pore refinement due to hydration, as the duration of the tensile test is short (less than 10 min). As tensile strain increases, micro-cracks form progressively within the ECC matrix and cause the reduction in conductivity with increasing strain.

To highlight the sensing capability of the composite, the conductivity of specimen DB2 is, for illustrative purposes, plotted against the tensile strain in Figure 10a together with the corresponding stress-strain relation. The conductivity is presented at selected spot frequencies
and the response near to the cusp point frequency, $f_c$, (5kHz) is highlighted with data markers. In all curves, only every 10th data marker is highlighted for reasons of clarity. This figure clearly shows the dependence of bulk conductivity on both tensile strain and test frequency. As the strain increases to ~0.15%, a reduction in conductivity is evident as micro-cracks begin to develop within the ECC matrix and the dominant conduction path at this stage would be via the uncracked portion. This is then followed by a rapid reduction in conductivity with increasing strain, resulting in approximately a fivefold decrease in value. To investigate the sensitivity of the composite to mechanical strain, the bulk conductivity, $\sigma(\omega)$, is converted to its reciprocal (bulk resistivity, $\rho(\omega)$) and then used to calculate the fractional change in resistivity (FCR) which is defined as (Wen and Chung, 2003; Ranade et al., 2014; Han et al., 2015; Chia and Huang, 2017; Ozbulut et al., 2018; Yang et al., 2018; Yoo et al., 2019),

$$\text{FCR} = \frac{\rho_f - \rho_i}{\rho_i}$$

(8)

where, $\rho_f$ is the bulk resistivity at strain $\varepsilon_f$ after the start of the test and $\rho_i$ is the initial bulk resistivity (i.e. at zero strain).

Figure 10b presents the FCR values for the four dog-bone specimens over the entire frequency range plotted against the fractional change in dimension (or strain), together with the stress-strain response measured from individual dog-bone specimens. As before, for reasons of clarity, only 10th data marker is highlighted in all curves and the FCR is presented to highlight its sensitivity to frequency. It is interesting to note from the Figure that by presenting the resistivity data in a dimensionless format (viz, the fractional change in resistivity versus the fractional change in dimension), this virtually removes the dependence of FCR on frequency and the resistivity curves all collapse onto a unique curve. It is evident that all specimens exhibit a quasi-linear increase in FCR with strain, with a notable increase.
in slope with increasing strain. The slope in specimen DB3 is shown to increase more appreciably at strain levels \( > \sim 3\% \), which coincides with the increase in the post-cracking stiffness shown in the figure reflecting the slip hardening behaviour resulting from abrasion damage on the fibre surfaces as the fibres being pulled out from the ECC matrix (Redon et al., 2001). This figure also indicates that the FCR of specimens DB2 and DB3 is very similar and consistently greater than the other two counterparts, which also display similarity in response. The FCR values at 4\% strain range between 2.4 and 4.2, which are comparable to the values reported by Ranade et al. (2014) for the high-volume fly-ash ECC mix and are lower than those for the moderate-volume fly-ash ECC mix reported in the same paper.

By plotting the FCR against the strain as in Figure 10b, distinct regions can be delineated in Figure 10c and denoted I–IV: (I) up to \( \sim 0.3\% \); (II): 0.3\%-1\%; (III): 1%-3\%; and (IV): >3\%, with the slope values highlighted. Considering the variations in FCR during the strain-hardening discussed above, it is interesting to note that this is not evident in the stress-strain responses of the four dog-bone specimens presented in Figure 10b, with all specimens displaying a comparable overall stiffness during the strain-hardening. As the electrical response of the specimens is affected by the presence of the micro-cracks, the apparent difference may thus originate from the distribution of the micro-cracks during loading. Attention thus focused on the number and width of the individual micro-crack during loading.

Crack Mapping during Tensile Loading

To corroborate electrical measurements regarding micro-cracking, Figures 11a and 12a present the representative longitudinal strain maps for, respectively, specimens DB3 and DB4 obtained using the DIC technique at every 0.5\% strain increment. The crack width distributions measured at 4\% strain and the corresponding best-fit lognormal distribution curves for both specimens are plotted in Figures 11b and 12b, respectively, with the best-fit
lognormal distribution curves at 1% strain increment presented in Figures 11c and 12c to aid data interpretation. It is apparent from the crack width distribution of specimen DB3 displayed in Figure 11c that there is a significant increase in the number of cracks at 3% strain, whereas the average crack width over this strain range remains relatively constant. The combined effect of this causes the displacement of the crack width distribution upwards; thereafter, there is a notable increase in the average crack width from $\sim$47$\mu$m at 3% strain to 65$\mu$m at 4.9% strain, thereby causing the distribution curve shown in Figure 11c to displace to the right. This trend agrees well with the strain maps depicted in Figure 11a, which displays an increasing number of near parallel bands of localised strain with a relatively constant colour range over the initial 3% strain, followed by a similar pattern of strain maps but with a notable increase in value. It could be inferred from the evolution of the crack pattern obtained that the increase in FCR for specimen DB3 over the initial 3% strain is attributed primarily to micro-crack formation whereas the increase in both values thereafter is due to the widening of existing micro-cracks.

With reference to the evolution of crack pattern of specimen DB4 (see Figures 12a, c), it is evident that micro-crack formation occurs constantly throughout the loading, from 2 fully-developed micro-cracks at 1% strain to 15 cracks at 4% strain, which is also accompanied by a notable increase in the average crack width. This would indicate that the predominant mechanism responsible for the increase in FCR presented previously in Figure 10b is due to progressive crack formation, with a significant contribution of crack width increase at strain levels $>$3%. It is interesting to note from both specimens that although they had more and less the same number of cracks ($\sim$15 cracks), with average crack width of $\sim$50$\mu$m at 4% strain, these two specimens attained different FCR values, with those of specimen DB3 being approximately twice those of specimen DB4. This would suggest the need for using the
actual crack width distribution to allow accurate calculation of FCR and work is continuing on this aspect.

**Conclusions**

Multiple testing methodology utilising electrical impedance measurements, mechanical testing and detailed image analysis has been employed to investigate the piezo-impedance properties of an engineered cementitious composite under direct tension, with the impedance measurements also employed to provide complementary information on the influence of on-going hydration. The following conclusions can be drawn from the work presented:

1. A distinct impedance response was observed at all stages during the hydration process. Increasing hydration is shown to result in an overall increase in impedance and a better definition of the high-frequency arc which reflects the development in the pore structure with time. A progressive increase in bulk resistance is evident during the entire curing period reflecting on-going hydration and pozzolanic reaction.

2. Dispersion in polarization is shown to result in a reduction in relative permittivity with increasing frequency and a corresponding increase in conductivity. Ongoing hydration and pozzolanic reaction are shown to have a negligible influence on the relative permittivity at the upper frequency limit (i.e. 1MHz).

3. Multiple micro-crack formation is shown to increase the sample impedance markedly and result in a progressive displacement and enlargement in the radius of the bulk arc when presented in the Nyquist format.
(4) It is shown that the relative permittivity at the high-frequency end (i.e. 1MHz) is sensitive to tensile straining, while virtually insensitive to on-going hydration. This parameter could, therefore, be exploited as a means of distinguishing changes in electrical properties due to on-going hydration and changes in electrical properties due to damage within the composite, when the matrix remains in a fully saturated condition.

(5) The composite sensitivity to strain is affected by the distribution of the micro-cracks. Larger FCR values were obtained from specimens that exhibit greater crack widths.

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Notation

\( F_{28} \) 28 d compressive strength (50 mm cube)

\( F_{90} \) 90 d compressive strength (50 mm cube)

\( f_c \) cusp-point frequency (Hz)

\( f_{cr} \) first crack tensile stress (MPa)

\( f_{tu} \) tensile strength (MPa)

\( FCR \) fractional change in resistivity

\( g_p \) geometrical constant for the prism specimen (=88.48/m)

\( g_{db} \) geometrical constant for the dog-bone specimen (=245.28/m)

\( R_{db,28} \) 28 d electrical resistance for the dog-bone specimen (\( \Omega \))

\( R_{p,28} \) 28 d electrical resistance for the prism specimen (\( \Omega \))

\( Z(\omega) \) electrical impedance (\( \Omega \))

\( Z'(\omega) \) resistive (real) component of impedance (\( \Omega \))

\( Z''(\omega) \) reactive (imaginary) component of impedance (\( \Omega \))

\( \varepsilon_t \) tensile strain at time \( t \) after the start of the test

\( \varepsilon_{tu} \) tensile strain capacity

\( \phi_{r,i} \) initial volume at the start of the test

\( \phi_{cr} \) volume fraction of the micro-cracks at strain \( \varepsilon_t \)

\( \phi_{r,t} \) total volume at strain \( \varepsilon_t \) after the start of the test

\( \kappa_0 \) permittivity of free space (8.854×10\(^{-12}\) Farads/m)

\( \kappa'_r(\omega) \) relative permittivity

\( \kappa_{r,i} \) initial bulk relative permittivity of the composite (at \( \varepsilon_t = 0 \))

\( \kappa_{cr} \) average relative permittivity of the micro-cracks

\( \kappa_{r,t} \) bulk relative permittivity of the composite at strain \( \varepsilon_t \) after the start of the test
\( \sigma(\omega) \) bulk conductivity (S/m)

\( \rho(\omega) \) bulk resistivity (\( \Omega \)m)

\( \rho_{28} \) 28 d electrical resistivity (\( \Omega \)m)

\( \rho_i \) initial bulk resistivity (at \( \varepsilon_t = 0 \)) (\( \Omega \)m)

\( \rho_t \) bulk resistivity at strain \( \varepsilon_t \) after the start of the test (\( \Omega \)m)

\( \omega \) angular frequency

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**Table captions**

**Table 1.** ECC mix proportions (in kg/m$^3$).

**Table 2.** Oxide analysis of materials (+ = not determined).

**Table 3.** Summary of mechanical properties. The number in brackets is the coefficient of variation for results (in %).
Table 1. ECC mix proportions (in kg/m³).

|     | C  | FA | S  | HRWR | PVA |
|-----|----|----|----|------|-----|
|     | 454| 818| 273| 4.54 | 26  |

Notes: C: CEM I 52.5N; FA: Fly-ash; S: Silica sand; HRWR: High range water reducer; and PVA: Polyvinyl alcohol fibres.

Table 2. Oxide analysis of materials (+ = not determined).

| Chemical analysis | PC   | Fly-ash | Silica sand |
|-------------------|------|---------|-------------|
| SiO₂              | 19.9 | 52.7    | 98.8        |
| Al₂O₃             | 4.8  | 26.6    | 0.21        |
| Fe₂O₃             | 3.1  | 5.6     | 0.09        |
| K₂O               | +    | +       | 0.03        |
| CaO               | 62.4 | 2.4     | +           |
| MgO               | 2.2  | 1.2     | +           |
| Na₂O equivalent   | 0.54 | 1.7     | +           |
| SO₃               | 3.0  | 0.3     | +           |
| Free CaO          | +    | 0.03    | +           |
| Total phosphate   | +    | 0.5     | +           |
| Loss on Ignition (LOI) | + | <2.0   | 0.14        |

Physical properties

| Specific gravity | 3.15 | 2.20 | 2.65 |
| Surface area (m²/kg) | 375 | 1300 | +    |
| Fineness (% retained on 25μm) | + | <25  | +    |
| Size distribution (μm) and cumulative retained (%) | |
| 500               | +    | +    | 0.1  |
| 355               | +    | +    | 0.5  |
| 250               | +    | +    | 1.5  |
| 180               | +    | +    | 6.0  |
| 125               | +    | +    | 46.0 |
| 90                | +    | +    | 83.0 |
| 63                | +    | +    | 96.5 |
Table 3. Summary of mechanical properties. The number in brackets is the coefficient of variation for results (in %).

| $\varepsilon_{tu}$ (%) | $f_{cr}$ (MPa) | $f_{tu}$ (MPa) | $F_{28}$ (MPa) | $F_{90}$ (MPa) |
|------------------------|----------------|----------------|----------------|----------------|
| 3.91                   | 2.96           | 4.38           | 51.2           | 63.7           |
| (19.1)                 | (10.7)         | (10.0)         | (1.94)         | (4.40)         |
Figure Captions

**Figure 1.** Schematic of testing arrangement for two-point measurements using (a) embedded perforated plate electrodes and (b) surface applied (silver-coated) wire electrodes.

**Figure 2.** (a) Test setup and (b) close-up of measurement during uniaxial tensile testing.

**Figure 3.** (a) Schematic of complex impedance response of a cementitious system and the idealised (b) conductivity and (c) relative permittivity dispersion curves.

**Figure 4.** Influence of curing on the electrical properties of prisms P1–P3 over the initial 90 days curing: (a) impedance response; (b) variation in cusp-point frequency, $f_c$; (c) relative permittivity versus frequency; and (d) conductivity versus frequency. The solid markers in (c) and (d) indicate the permittivity/conductivity corresponding to the cusp point of the impedance response presented in (a).

**Figure 5.** Variation in impedance response with tensile strain obtained from specimens (a) DB1; (b) DB2; (c) DB3; and (d) DB4.

**Figure 6.** Schematic of possible conduction pathways in (a) uncracked, and (b) cracked ECC matrix showing possible bridging pathways.
Figure 7. Relative permittivity versus frequency at selected strain levels for specimens (a) DB1; (b) DB2; (c) DB3; and (d) DB4. The cusp point frequency is presented as a solid marker.

Figure 8. (a) Idealized representation of ECC before and after cracking; and (b) variation in relative permittivity of the micro-cracks assuming a series mixing law (equation (6)).

Figure 9. Conductivity versus frequency response under tensile load for specimens: (a) DB1; (b) DB2; (c) DB3; and (d) DB4. The cusp point frequency is presented as a solid marker.

Figure 10. (a) Variation in the bulk conductivity of specimen DB2 under varying tensile strains; (b) the fractional change in resistivity for specimens DB1–DB4 under varying tensile strains; and (c) representation of the fractional change in resistivity of specimen DB4 into contiguous linear segments. All curves are plotted alongside the stress-strain responses.

Figure 11. (a) Progressive development of longitudinal strains on the front face of specimen DB3; (b) observed and fitted crack width distribution at 4% strain from visual observations; and (c) fitted crack width distributions at varying strain levels.

Figure 12. (a) Progressive development of longitudinal strains on the front face of specimen DB4; (b) observed and fitted crack width distribution at 4% strain from visual observations; and (c) fitted crack width distributions at varying strain levels.
Figure 1
Figure 2
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Figure 6
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Figure 8
Figure 9
Figure 10.
Figure 11
Figure 12