

Non-destructive monitoring of moisture at the steel-concrete interface with embedded RFID MEMS sensors

Jia Xiang Liew

Department of Civil and Environmental Engineering

Imperial College London

March 2023

A thesis submitted in fulfilment of the requirements for the degree of Doctor Philosophy and Diploma of Imperial College London

Declaration of originality

I hereby declare that this thesis, submitted for the degree of Doctor of Philosophy and Diploma of Imperial College London is the result of my own investigations. Any information derived from the work of others, published or unpublished, has been acknowledged in the text and a list of references is given. I hereby certify that the work presented in this thesis has not been submitted in any form to this or any other institution, for any degree, diploma or other qualification.

The copyright of this thesis rest with the author and is made available under a Creative Commons Attribution Non-Commercial No Derivatives license. Researchers are free to copy, distribute or transmit the thesis on the condition that they attribute it, that they do not use it for commercial purposes and that they do not alter, transform or build upon it. For any reuse or redistribution, researchers must make clear to others the licence terms of this work.

Jia Xiang Liew

March 2023

Abstract

The moisture state of concrete and entrapped air voids at the steel-concrete interface are important influences on the corrosion of reinforced concrete, but the detail of these relationships is unclear. This study investigates the suitability of monitoring relative humidity at the steel-concrete interface with radio-frequency identification (RFID) microelectromechanical systems (MEMS) sensors in customized polylactic acid enclosures. This involved prototyping and calibrating enclosure designs, determining the influence of cementitious material on wireless communication and embedding RFID MEMS sensors in concrete specimens. It was shown for the first time that relative humidity at the steel-concrete interface can be monitored for samples with different curing ages, nominal cover depths and water to cement ratio, demonstrating the suitability of such a device for laboratory-based research.

The method was used to monitor relative humidity changes in air voids in specimens subjected to wetting and drying. The effects of sample preparation, void size and microcracks at the paste-void interface (characterised by SEM-BSE) induced by drying conditions were also examined. It was shown that RFID MEMS sensors were capable of monitoring relative humidity changes in air voids with faster rates of saturation for smaller voids compared to larger voids.

The durability and reliability of sensor components were also investigated. RFID MEMS sensors were subjected to 1 year of cyclic wetting and drying, 7 months of embedment in high and low-strength concrete, 3 months of exposure to 3% wt. NaCl salt solution and 100% RH environment. The impact of cementitious material on sensor components was studied with SEM-BSE and EDS. Surface morphology of MEMS sensors was also examined and related to relative humidity measurements to explain changes in performance. Results demonstrated that similar measurements were obtained when compared to unexposed sensors; however, performance was affected after exposure to 3% wt. NaCl salt solutions for 63 days.

Acknowledgements

I am forever grateful to my supervisors, Professor Nick Buenfeld and Professor Hong Wong for their continuous encouragement, profound knowledge, guidance and support over the last four years. It has been a great honour to have the opportunity to pursue this PhD under their supervision.

This PhD work would not have been completed without the support, advice and encouragement of the following individuals:

- Dr Marcus Yio for his valuable comments, discussions and training for many experimental techniques in the laboratory;
- Mr Andrew Morris for his dedicated help with the laboratory work ;
- My colleagues: Alalea, Kai, Xi, Sam, Roshni, Shiyong, He, Alexander, Barnaby, Adam, Kaine, Terry, Teijun, Meng, Tsz Chun, Maria, Mehrdad, for the ideas, lab help and funny jokes;
- Friends in the department and college: Sihua, Qiming, Deema, Javier, Osama, Maria, Kamil, Fahmy, Eduardo, He-in, Petrina, Hanyu, Cheng-Yu, and many others for their motivation and companionship;
- Sarah Willis, Fionnuala Ni Dhonnabhain, Angela Frederick and many other staffs from the Department of Civil and Environmental Engineering, Central Library, International Office and Student Hub for their assistance.

Last but not least, I would like to express my special gratitude and thanks to my parents and family for the countless sacrifices they have made throughout these years. I wish I can let them know how much I love them and how much I appreciate the things they have done for me.

Notation

Roman letters

BSE	Backscattered electron
C	Concrete
CSH	Calcium Silicate Hydrate
C ₂ S	2CaO.SiO ₂
C ₃ S	3CaO.SiO ₂
C ₃ A	3CaO.Al ₂ O ₃
C ₄ AF	4CaO.Al ₂ O ₃ .Fe ₂ O ₃
EDS	Energy dispersive X-ray spectroscopy
Fe	Iron
HF	High frequency
i	Cumulative absorbed water per unit area of inflow face (g/m ²)
M	Mortar
m	Mass of sample
MEMS	Microelectromechanical systems
MSA	Maximum size of aggregate (mm)
P	Paste
PCB	Printed circuit boards
pH	Potential of hydrogen
PLA	Polylactic acid
PVC	Polyvinyl chloride

RFID	Radio-frequency identification
RH	Relative humidity (%)
ROI	Region of interest
S	Sorptivity coefficient ($\text{g/m}^2 \cdot \text{min}^{1/2}$)
SCMs	Supplementary cementitious materials
SEM	Scanning electron microscopy
Si	Silicon
SiC	Silicon carbide
SNR	Signal-to-noise ratio
SSD	Saturated-surface dry
t	Elapsed time
UHF	Ultra-high frequency
UV	Ultraviolet
w/b	Water/binder ratio
w/c	Water/cement ratio

Greek letters

ρ	The density of water (g/mm^3)
$\emptyset$	Diameter (mm)

Contents

Declaration of originality	2
Abstract	3
Acknowledgements	4
Notation	5
Contents	7
List of Tables	14
List of Figures	15
Chapter 1 Introduction	24
1.1 Background	24
1.2 Objectives and scope	25
1.3 Thesis outline	25
Chapter 2 Literature review	27
2.1 Reinforcement corrosion	27
2.2 Steel concrete interface	29
2.2.1 Passive film	29
2.2.2 Concrete microstructure and chemistry	29
2.2.3 Macroscopic voids	30
2.3 Monitoring techniques	30
2.3.1 In-situ	31
2.3.1.1 Capacitance	31
2.3.1.2 Resistance	32
2.3.1.3 Fiber Optic and Bragg grating	34
2.3.1.5 Microwave and Radio Frequency	35
2.3.1.6 Nuclear magnetic resonance	36
2.3.1.7 Piezoelectric sensors	37

2.3.2 Ex-situ	38
2.3.2.1 X-ray tomography	39
2.3.2.2 Neutron imaging	40
2.3.2.3 Nuclear magnetic resonance imaging	41
2.4 Discussion	42
2.5 Impact on reliability and performance of embedded sensors.....	45
2.5.1 Wireless communication.....	46
2.5.2 Accuracy	47
2.5.3 Durability	48
2.5.4 Enhancements	51
2.6 Chapter Summary	52
Chapter 3 Methodology	53
3.1 Materials and mix proportion.....	53
3.1.1 Materials	53
3.1.2 Mix proportions	55
3.1.3 Sample conditioning	56
3.2 Scanning electron microscope (SEM).....	56
3.2.1 Principle	57
3.2.2 Sample preparation	58
3.2.2.1 Cementitious material	58
3.2.2.2 RFID MEMS sensor.....	60
3.2.3 Instrument	61
3.3 Stereomicroscope	62
3.3.1 Principle	62
3.3.2 Olympus BX51 stereomicroscopy	63
3.3.3 Olympus SZX10 stereomicroscopy	64
3.3.4 Adjusting colour threshold for degradation analysis	65

3.4 Chapter Summary	67
Chapter 4 Developing a novel non-destructive method to monitor relative humidity at the steel-concrete interface using RFID-MEMS sensors	68
4.1 Introduction.....	68
4.2 Sensor selection	69
4.2.1 Microchip Technology HS1101LF capacitive humidity sensor	70
4.2.1.1 Sensor design	70
4.2.2 Renesas HS3000 humidity sensor.....	72
4.2.2.1 Sensor design	72
4.2.2.2 Hardware and electronic components	73
4.2.3 Texas Instruments HDC2010 humidity and temperature sensor	73
4.2.3.1 Sensor design	73
4.2.3.2 Hardware and electronic components	74
4.2.4 Sensirion SHT4x relative humidity and temperature sensor.....	75
4.2.4.1 Sensor design	75
4.2.4.2 Hardware and electronic components	75
4.2.5 STMicroelectronics HTS221 capacitive digital relative humidity and temperature sensor.....	76
4.2.5.1 Sensor design	76
4.2.5.2 Hardware and electronic components	77
4.2.6 Summary	77
4.3 Prototype and design.....	79
4.3.1 Commercially purchased enclosures.....	79
4.3.1.1 Experimentation.....	80
4.3.1.2 Preliminary result and discussion	81
4.3.2 3D printing	84
4.3.2.1 Software	84
4.3.2.2 Sample preparation	84

4.3.2.3 Fused deposition modelling technique and design procedures	85
4.3.3 Second Prototype	87
4.3.4 Final design.....	89
4.3.4.1 Loading and deflection tests	90
4.4 Testing precision and accuracy of MEMS sensors	91
4.4.1 Results.....	92
4.4.1.1 Accuracy of MEMS	92
4.4.1.1.1 Increasing relative humidity.....	92
4.4.1.1.2 Decreasing relative humidity	94
4.4.1.2 Rate of response	95
4.4.1.2.1 Increasing relative humidity.....	95
4.4.1.2.2 Decreasing relative humidity	96
4.4.2 Discussion	97
4.4.2.1 Effects of moisture on accuracy, precision and response of RFID MEMS sensor	97
4.5 Wireless communication range.....	99
4.5.1 Results.....	100
4.5.1.1 Effects on wireless communication range.....	100
4.5.1.1.1 Influence of aggregate content and degree of saturation.....	100
4.5.1.1.2 Directional placement	102
4.6 Transmitted power analysis	103
4.6.1 Results.....	105
4.6.1.1 Effects on transmitted power	105
4.6.2 Discussion	107
4.6.2.1 Influence of directional placement, moisture and aggregate content on wireless communication and transmitted power	107
4.7 Enclosure design	108
4.7.1 Results.....	109

4.7.1.1 Influence of enclosure design.....	109
4.7.1.2 Discussion.....	110
4.8 Monitoring relative humidity.....	111
4.8.1 Results.....	112
4.8.2 Discussion.....	115
4.9 Conclusions.....	115
Chapter 5 Examining the reliability and performance of RFID-MEMS sensors exposed to cementitious materials and simulated marine environments.....	117
5.1 Introduction.....	117
5.2 Preliminary studies.....	118
5.2.1 Materials and mix proportions	118
5.2.2 Samples	119
5.2.2.1 Commercially purchased enclosure	119
5.2.2.2 Customized PLA enclosure.....	119
5.2.2.3 Olympus BX51 stereomicroscopy	119
5.2.3 Results and discussion	120
5.2.4 Limitations and improvements.....	121
5.3 Experimental.....	121
5.3.1 Materials and mix proportions	121
5.3.2 Samples	122
5.3.3 Simulated marine environments.....	122
5.3.4 Fatigue of MEMS sensors.....	123
5.3.5 Performance test.....	124
5.3.6 Olympus BX51 stereomicroscopy	124
5.3.7 Backscattered electron microscopy (BSE)/Energy Dispersive Spectroscopy (EDS)	124
5.4 Results.....	126
5.4.1 Surface morphology vs. performance	126

5.4.1.1	Influence of cementitious materials	126
5.4.1.2	Direct exposure to 100% RH and 3% wt. NaCl.....	128
5.4.1.3	Fatigue of MEMS sensors.....	130
5.4.2	Particulate and contamination analysis	132
5.4.3	Area of degradation.....	134
5.4.4	Ageing of electronic components.....	135
5.4.4.1	Capacitor	135
5.4.4.2	Resistor	137
5.5	Discussion	140
5.5.1	Durability and performance of MEMS sensors	140
5.5.2	The effects of particulate contamination on silicon thin films.....	141
5.5.3	Durability of electronic components.....	142
5.6	Conclusions.....	143
Chapter 6 Characterising moisture state of air voids in cement-based materials subjected to wetting-drying using RFID-MEMS sensors.....		144
6.1	Introduction.....	144
6.2	Theoretical background	145
6.3	Simulating air voids at the steel-concrete interface	147
6.3.1	Methods used to simulate air voids.....	148
6.3.2	Limitations	150
6.3.3	Influence on transport properties	152
6.3.3.1	Samples	152
6.3.3.2	Fluorescence imaging technique.....	153
6.3.3.3	Water absorption.....	153
6.3.3.4	Result and discussion	154
6.3.3.5	Summary	158
6.4	Experimental.....	158

6.4.1 Materials and mix proportions	158
6.4.2 Samples	158
6.4.3 Water absorption	159
6.4.4 Water desorption	160
6.4.5 Monitoring internal relative humidity	161
6.4.6 Olympus SZX10 stereomicroscopy	162
6.4.7 Backscattered electron microscopy (BSE).....	162
6.4.7.1 Analysis.....	163
6.5 Results.....	163
6.5.1 Cumulative absorption vs. $t^{1/2}$ plots	163
6.5.2 Rate of increase in relative humidity	166
6.5.3 Cumulative desorption	168
6.5.4 Rate of decrease in relative humidity	170
6.5.5 Surface morphology of air voids.....	172
6.6 Discussion	177
6.6.1 Effect of geometrical size of entrapped air voids on absorption rates	177
6.6.2 Comparing internal relative humidity with cumulative absorption measurements.....	178
6.6.3 Comparing internal relative humidity with cumulative desorption measurements.....	179
6.7 Conclusions.....	179
Chapter 7 Conclusion and Future Work	181
References.....	185

List of Tables

Table 2-1 Overview of technologies and their potential for monitoring moisture at the steel-concrete interface.	43
Table 2-2 Mixed flowing gas (MFG) test parameters (Broas et al. 2015).	51
Table 3-1 Oxide composition of CEM I	54
Table 3-2 Aggregate properties.....	54
Table 4-1 Design considerations.....	70
Table 4-2 Overview of sensor technologies and potentials for monitoring relative humidity at the steel interface	78
Table 4-3 Mix proportions.....	80
Table 5-1 Mix proportions.	118
Table 5-2 Mix proportions.	121
Table 6-1 Mix proportions.	148
Table 6-2 Geometrical dimensions of air voids.	149
Table 6-3 Mix proportions.	158

List of Figures

Figure 2-1 A typical structure of a capacitive humidity sensor (Raunima et al. 2019).....	32
Figure 2-2 Illustration of the multi electrode array with a) a schematic design and b) distribution at different cover depths (McCarter et al. 2012).....	33
Figure 2-3 Schematic design of experimental setup to obtain relative humidity measurements from polymer coated FBG sensor (Yeo, Eckstein, et al. 2006).	34
Figure 2-4 MEMS microcantilever sensor with a) a schematic design and b) functionality of the expanded water vapour sensitive polymer (Norris, Saafi, and Romine 2008).....	35
Figure 2-5 Schematic of calibration system to obtain direct relationships between applied oscillation frequency and moisture content (Sokoll and Jacob 2014).	36
Figure 2-6 Internal components of NMR sensor (P. F. de J. Cano-Barrita et al. 2009).....	37
Figure 2-7 Internal components of the piezoelectric sensor (Kong et al. 2013)	38
Figure 2-8 Overview of experimental setup to examine water transport in concrete (Oesch et al. 2019). .	39
Figure 2-9 Experimental setup to analyze moisture migration in concrete at high temperatures (Duati et al. 2018)	40
Figure 2-10 Experimental setup for ¹ H nuclear magnetic resonance imaging to examine moisture content (P. F. Cano-Barrita and Díaz-Díaz 2021)	41
Figure 2-11 Radio attenuation per metre in concrete (Daly, Melia, and Baldwin 2010).....	47
Figure 2-12 Changes in capacitance over time for capacitors exposed to different environmental conditions (Donahoe et al. 2006).	48
Figure 2-13 Images of embedded a) HF RFID sensors and b) UHF RFID sensors (Strangfeld, Johann, and Bartholmai 2019).	49
Figure 2-14 Photographs of sensors a) with epoxy resin and b) without epoxy resin (Strangfeld, Johann, and Bartholmai 2019).	49
Figure 2-15 Photographs of capacitive humidity sensors a) with epoxy resin and b) without epoxy resin (Strangfeld, Johann, and Bartholmai 2019).	50
Figure 2-16 Overview of MEMS microphone with a) schematic and b) SEM image of package structure (Broas et al. 2015).....	50
Figure 2-17 Normalized fracture force on MEMS microphone after the exposure to mixed flowing gas test (Broas et al. 2015).	51

Figure 3-1 Grading curves for Thames valley sand and gravel.	54
Figure 3-2 Sample designation.	55
Figure 3-3 Schematic drawing of scanning electron microscope (Kwiecińska, Pusz, and Valentine 2019)	57
Figure 3-4 Equipment for vacuum epoxy impregnation.	59
Figure 3-5 Equipment for deep impregnation.	59
Figure 3-6 Cement paste specimen with a 6.5 mm diameter air void placed a) in 65 × 65 × 30 mm mould for encasing and b) vacuum impregnated in a pressure chamber at 3 bars.	60
Figure 3-7 Extracting 10 × 50 × 25 mm block samples marked with dotted lines to examine void-paste interface with SEM-BSE.	60
Figure 3-8 Hitachi TM4000 Plus scanning electron microscope.	61
Figure 3-9 Schematics of common main objective (CMO) and Greenough optical systems (Miyake et al. 2019).	62
Figure 3-10 Olympus BX51 stereomicroscope.	63
Figure 3-11 Olympus SZX10 stereomicroscope.	64
Figure 3-12 The influence of luminance values on estimated degradation area.	66
Figure 4-1 HS1101LF relative humidity sensor manufactured by Humirel with a) schematics and b) sensing principle.	71
Figure 4-2 Schematics of Humidity Sensor PICtail™ Demo board with the dimensions of 65 × 50 mm attached to a PICKit™ Flash starter kit to evaluate relative humidity measurements.	72
Figure 4-3 HS 3000 humidity sensor manufactured by Renesas with a) schematics and b) sensing principle.	72
Figure 4-4 Schematics of Renesas SDAH01 Evaluation Kit with the overall dimensions of 44 × 32 mm and HS 3000 humidity sensor attached.	73
Figure 4-5 Schematic of HDC2010 low-power digital relative humidity sensor manufactured by Texas instruments.	74
Figure 4-6 Schematics of Texas TIDA-01477 Humidity and Temperature Sensor Node with the overall dimensions of 76.2 × 38.1 mm and HDC2010 humidity sensor attached.	74
Figure 4-7 Schematics of SHT4x relative humidity and temperature sensor manufactured by Sensirion.	75
Figure 4-8 Schematics of Sensirion Smart Gadget with the overall dimensions of 75 × 32 mm and SHT4x humidity sensor attached.	76

Figure 4-9 HTS221 capacitive digital relative humidity and temperature sensor with a) schematics and b) sensing principle (J I Izpura 2001).....	76
Figure 4-10 Schematic of STMicroelectronics STEVAL-SMARTAG1 with the overall dimensions of 43 × 43 mm.	77
Figure 4-11 Schematics of 45 × 45 × 20 mm polyethylene terephthalate enclosure with five drilled one-millimetre diameter circular holes.	79
Figure 4-12 RFID MEMS sensors embedded in 100 × 100 × 100 mm concrete and mortar specimens a) wrapped in wet hessian fabric and b) placed in incubators containing ammonium nitrate (65% RH) and magnesium chloride (33% RH) saturated salts.	81
Figure 4-13 Measured changes in the relative humidity of samples in incubators containing saturated ammonium nitrate (65% RH) at 72 hours and magnesium chloride (33% RH) at 312 hours and 408 hours.	82
Figure 4-14 Measured changes in temperature of samples in incubators containing saturated ammonium nitrate (65% RH) at 72 hours and magnesium chloride (33% RH) at 312 hours and 408 hours.	82
Figure 4-15 Examining the enclosure-specimen interface of a) concrete and b) mortar after wet cutting with Buehler Delta automatic abrasive cutter.	83
Figure 4-16 Schematics of Ultimaker S5 pro.....	85
Figure 4-17 Schematics of the second prototype with the a) side and b) bottom view.....	88
Figure 4-18 Water bleeding into the enclosure during concrete pouring.....	88
Figure 4-19 Schematics of the final design with the a) side, b) bottom view and c) top view of the sample after 24 hours of curing.....	89
Figure 4-20 Comparing the developed method with commercially available reinforcement spacers for 50mm of cover on the left and 100mm of cover on the right.	90
Figure 4-21 Image illustrating test method used to determine the maximum deflection of PLA 3D printed enclosure a) with a 100 KN load cell attached and b) closer image of the test method.....	91
Figure 4-22 Conditioning incubators and enclosures a) with relative humidity examined by Fisherbrand Traceable thermometer and humidity monitor and b) for precision and accuracy testing with RFID MEMS sensors.....	92
Figure 4-23 Evaluating the accuracy of RFID MEMS by placing sensors in incubators containing saturated salts in the order of sodium chloride (75% RH), potassium hydrogen sulphate (86% RH) and potassium nitrate (93% RH).....	93

Figure 4-24 Evaluating the accuracy of RFID MEMS by placing sensors in incubators containing saturated salts in the order of potassium nitrate (93% RH), potassium hydrogen sulphate (86% RH) and sodium chloride (75% RH).	95
Figure 4-25 Examining the rate of response of RFID MEMS by placing sensors in incubators containing saturated salts in the order of sodium nitrate (75% RH), potassium hydrogen sulphate (86% RH), potassium nitrate (93% RH) salt solutions and moisture (100% RH).	96
Figure 4-26 Examining the rate of response of RFID MEMS by placing sensors in incubators containing saturated salts in the order of moisture (100% RH), potassium nitrate (93% RH), potassium hydrogen sulphate (86% RH) and sodium nitrate (75% RH).....	97
Figure 4-27 Components and structure of the sensing element a) without the influence and b) with the influence of stiction and moisture.	98
Figure 4-28 Test method to determine the maximum communication distance by placing RFID MEMS sensor a) below 50 and 100mm concrete and mortar specimens and b) at different orientations.....	100
Figure 4-29 Change in maximum communication range measured after submerging specimens in tap water.....	101
Figure 4-30 Change in maximum communication range measured by tilting RFID MEMS sensor away from RFID reader.....	103
Figure 4-31 Sectioning mortar and cement paste specimens with Isomet 1000 precision cutter of mortar.	104
Figure 4-32 Electrical schematics of ST STEVAL SMARTAG 1 with 2.5mm diameter wires soldered onto the R6 resistor and diode attached to a Solartron 7050 digital multimeter to monitor peak transmitted power.	104
Figure 4-33 Placement of mortar and cement paste specimens above RFID MEMS sensor to determine the maximum transmission power received.	105
Figure 4-34 Peak transmitted power received by RFID MEMS sensor measured at a) 97-105 mm, b) 105-130 mm and c) 135-145 mm communication distances.....	106
Figure 4-35 The influence of moisture and aggregate content on transmitted power at 13.56 MHz frequency.....	107
Figure 4-36 Directly exposing RFID MEMS sensors to 100% RH humidity.	108
Figure 4-37 Directly exposing RFID MEMS sensors with an enclosure to 100% RH humidity.	109
Figure 4-38 Monitoring changes of relative humidity for RFID MEMS sensors with and without enclosures.....	110

Figure 4-39 RFID MEMS sensor embedded in 3D printed enclosure with an abundance of space around sensor components and battery compartment.	111
Figure 4-40 Embedment of 3D printed enclosures a) in 100 mm diameter plastic moulds and b) schematic design of developed method in concrete specimens.	112
Figure 4-41 High strength concrete 14 days cured specimens with a) improper and b) proper compaction.	112
Figure 4-42 Monitoring changes of internal relative humidity at the steel-concrete interface with RFID MEMS sensors in low and high-strength concrete specimens with 10mm nominal cover depth during oven drying at 50°C.	113
Figure 4-43 Monitoring changes of internal relative humidity at the steel-concrete interface with RFID MEMS sensors in low and high-strength concrete specimens with 10, 25 and 50 mm nominal cover depth.	114
Figure 5-1 Microscopy image of MEMS sensor a) before embedment, b) 28 days in commercially purchased polyethylene terephthalate enclosures and c) 28 days in 3D printed PLA enclosure.	120
Figure 5-2 Retrieving RFID MEMS sensors from concrete samples after 7 months of embedment.	122
Figure 5-3 Exposing RFID MEMS sensors to 100% RH humidity and 3% wt. NaCl salt solution.	123
Figure 5-4 Exposing RFID MEMS sensors to wetting and drying cycles to examine fatigue.	123
Figure 5-5 a) Adjusting colour threshold of images obtained from stereomicroscope and b) estimating total area of degradation of silicon material.	124
Figure 5-6 Adjusting colour threshold of images a) obtained from backscattered electron microscopy and b) estimated area of degraded silicon material.	125
Figure 5-7 Examining coatings and solder joints of a) capacitors and b) resistors.	125
Figure 5-8 Analysing degradation site on silicon thin films.	126
Figure 5-9 Examining the surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 after embedding in low and high strength concrete for 7 months subjected to wetting and drying cycles.	127
Figure 5-10 Performance of RFID MEMS sensors after embedding in low and high strength concrete for 7 months subjected to wetting and drying cycles.	127
Figure 5-11 Examining the surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 after direct exposure to 100 % RH for 63 days.	128
Figure 5-12 Performance of RFID MEMS sensors after direct exposure to 100% RH for 63 days.	129

Figure 5-13 Examining the surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 after direct exposure to 3% wt. NaCl salt solution for 63 days.	129
Figure 5-14 Performance of RFID MEMS sensors after direct exposure to 3% wt. NaCl salt solution for 63 days.	130
Figure 5-15 Examining surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 subjected to 1 year of cyclic wetting and drying.....	131
Figure 5-16 Performance of MEMS sensors after being subjected to 1 year of cyclic wetting and drying.	131
Figure 5-17 Examining degradation of silicon thin film exposed to cementitious materials and simulated environments with SEM-BSE.....	133
Figure 5-18 Elemental analysis of degraded material after exposure to cementitious materials and simulated environments with SEM-EDS.	133
Figure 5-19 Estimating the total area of degradation of silicon thin films over time after direct exposure to 3% wt. NaCl salt solution and 100% RH for 2 months.	134
Figure 5-20 Examining capacitor after exposure to cementitious materials and simulated environments with SEM BSE.....	136
Figure 5-21 Elemental analysis of capacitor after exposure to cementitious materials and simulated environments with SEM-EDS.....	137
Figure 5-22 Examining soldering joints of resistor after exposure to cementitious materials and simulated environments with SEM BSE.	139
Figure 5-23 Elemental analysis of solder joints after exposure to cementitious materials and simulated environments with SEM-EDS.....	140
Figure 5-24 Figure to illustrate materials and structure of RFID MEMS sensor a) before and b) after degradation.....	142
Figure 6-1 Amount of compressed and saturated air in a spherical air void during capillary absorption at 20°C.....	147
Figure 6-2 Customized 50 × 50 × 50mm steel mould with a) 2mm diameter drilled holes and b) 1ml syringe with the needle inserted at the bottom of the base plate.....	149
Figure 6-3 Equipment and apparatus setup of Micromeritics AccuPyc II gas displacement pycnometer.	149
Figure 6-4 Surface morphology of simulated air voids and air voids imprinted with a 20 mm diameter stainless steel spoon on cement paste specimens.....	150

Figure 6-5 Cross-sectional view of 20% aggregate volume and 0.4 w/c mortar samples with a) 0.4 ml and b) 0.6 ml of air injected after 1 minute 30 seconds of vibration.	151
Figure 6-6 Cross-sectional view of 20% aggregate volume and 0.4 w/c mortar samples with a) 0.4 ml and b) 0.6 ml of air injected after 3 minutes of vibration.	151
Figure 6-7 Cross-section view of 30% aggregate volume and 0.5 w/c mortar samples with a) 0.6 ml and b) 0.8 ml of injected air after 1 minute 30 seconds of vibration.	151
Figure 6-8 Cross-section view of 50% aggregate volume mortar sample with 0.6 ml of injected air after 1 minute of vibration.....	152
Figure 6-9 Exposing cement paste specimen to water containing 1% wt. fluorescein.	153
Figure 6-10 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.35 w/c cement paste specimens without vibration.	154
Figure 6-11 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.35 w/c cement paste specimens with vibration.	155
Figure 6-12 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.4 w/c cement paste specimens without vibration.	155
Figure 6-13 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.4 w/c cement paste specimens with vibration.	155
Figure 6-14 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.45 w/c cement paste specimens without vibration.	156
Figure 6-15 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.45 w/c cement paste specimens with vibration.	156
Figure 6-16 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.5 w/c cement paste specimens without vibration.	156

Figure 6-17 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.5 w/c cement paste specimens with vibration.	157
Figure 6-18 Cumulative absorption of 3 day cured 0.35w/c, 0.4 w/c, 0.45 w/c and 0.5 w/c cement paste specimens with needle insertion and without insertion.....	157
Figure 6-19 Schematics of experimental setup for water absorption.....	160
Figure 6-20 Schematics of experimental setup for water desorption.....	160
Figure 6-21 Schematics of 3D printed PLA enclosure with a) top and b) side view.....	161
Figure 6-22 Images of a) STMicroelectronics STEVAL SMARTAG1 and b) ST25R3911B-DISCO RFID reader.	161
Figure 6-23 Testing leakage of developed technique to monitor the internal relative humidity of air voids.	162
Figure 6-24 Examples of microcracks at the paste-void interface examined with SEM/BSE after conditioning at 50°C and exposure to wetting and drying.....	163
Figure 6-25 Examples of cumulative water absorption plots for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C.....	165
Figure 6-26 Examples of cumulative water absorption plots for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.	166
Figure 6-27 Examples of internal relative humidity measurements for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C. ...	167
Figure 6-28 Examples of internal relative humidity measurements for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.	168
Figure 6-29 Examples of cumulative water desorption plots for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C.....	169
Figure 6-30 Examples of cumulative water desorption plots for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.	170
Figure 6-31 Examples of internal relative humidity measurements for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C. ...	171
Figure 6-32 Examples of internal relative humidity measurements for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.	172
Figure 6-33 Maximum crack length formed on surfaces of air voids before conditioning at 50°C for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	173

Figure 6-34 Examples of cracks forming on surfaces of air voids after conditioning at 50°C and exposure to wetting and drying for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	174
Figure 6-35 Maximum crack length formed on surfaces of air voids before conditioning at 50°C for 0.45 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	174
Figure 6-36 Examples of cracks forming on surfaces of air voids after conditioning at 50°C and exposure to wetting and drying for 0.45 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	174
Figure 6-37 Maximum crack length formed on surfaces of air voids before gentle drying at 55% RH for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	175
Figure 6-38 Examples of cracks forming on surfaces of air voids after gentle drying at 55% RH and exposure to wetting and drying for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	175
Figure 6-39 Maximum crack length formed on surfaces of air voids before gentle drying at 75% RH for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	175
Figure 6-40 Examples of cracks forming on surfaces of air voids after gentle drying at 55% RH and exposure to wetting and drying for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.	176
Figure 6-41 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.5 w/c 3 days cured cement paste specimens after conditioning at 50°C and exposure to wetting and drying.	176
Figure 6-42 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.45 w/c 3 days cured cement paste specimens after conditioning at 50°C and exposure to wetting and drying.	176
Figure 6-43 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.5 w/c 3 days cured cement paste specimens after conditioning at 55% RH and exposure to wetting and drying.	177
Figure 6-44 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.5 w/c 3 days cured cement paste specimens after conditioning at 75% RH and exposure to wetting and drying.	177

Chapter 1 Introduction

1.1 Background

Reinforced concrete is the most heavily used construction material in the world. However, structures are prone to reinforcement corrosion which is a costly and widespread deterioration process (2.1).

In particular, the moisture state of the bulk concrete and entrapped air voids at the surface of the steel can critically influence corrosion initiation and substantially affect corrosion rates. For example, carbonation can reach a maximum rate at a relative humidity between 50 and 70 % (Wierig 1984). Lower critical chloride contents can be observed at a relative humidity range of 80 to 95 % (Song, Kong, and Liu 2007). In addition, partially saturated interfacial voids containing chlorides can increase the risk of corrosion and buffer the pH during early pitting corrosion. However, these relationships are inadequately understood with apparent conflicting evidence and opinions presented in the literature (2.2).

A laboratory-based method for monitoring the moisture state of the bulk concrete and entrapped air voids at the steel-concrete interface would help to clarify the aforementioned relationships. If the method could be applied for in-situ monitoring on real structures a more reliable assessment of corrosion risk can be provided. Moreover, reasonable sampling regimes, communication schedule, sensing range, accuracy and repeatability should be considered to obtain valuable information regarding the electrochemical behaviour during corrosion initiation. However, existing methods do not provide a direct measurement and evaluation of the steel-concrete interface (2.3).

With the aforementioned issues, it is clear that there is a need to develop alternative methods to monitor moisture content. This research explores the potential of radio-frequency identification (RFID) microelectromechanical systems (MEMS) sensors for these applications including the considerations of scientific principles, sensor components, embedment methods and applications (2.3). RFID MEMS sensors could be embedded in customized enclosures to monitor steel reinforcement with a minimum diameter of 8 mm and provide the ability for quantification within the millimeter range to characterize macroscopic interfacial voids. Furthermore, the suitability of monitoring relative humidity at the steel-

concrete interface with RFID MEMS sensors will be investigated, the reliability of sensor components will be examined with SEM BSE and EDS and related to relative humidity measurements to explain changes in performance, relative humidity changes in simulated air voids will be characterized with gravimetric measurements to explain water absorption and desorption attributes.

1.2 Objectives and scope

The overall aim of this thesis is to develop a suitable and reliable non-destructive technique for monitoring changes in relative humidity at the steel-concrete interface based on RFID MEMS sensors. The moisture state of air voids at the steel-concrete interface is of particular interest.

This will be achieved by the following objectives:

- To develop reliable enclosures to contain and prevent particulate contamination and degradation of RFID MEMS sensors while complying with the British standards for reinforcing bar spacers.
- To examine the suitability of RFID MEMS sensors for monitoring the relative humidity at the steel interface.
- To evaluate the reliability of RFID MEMS sensors exposed to cementitious materials, moisture and corrosive environments
- To investigate the impact of cycling wetting and drying on the on long-term performance of RFID MEMS sensors
- To apply the developed technique to characterize the moisture state of air voids with RFID MEMS sensors

1.3 Thesis outline

This thesis is divided into eight chapters. Chapter 1 outlines the background, objectives, and scope of the study. Chapter 2 consists of a literature review on subjects relevant to the scope of this thesis. Chapter 3

presents the experimental methodology including the materials, sample preparation and instrumentation. The main findings of this research are presented in the following three chapters (4-6).

MEMS sensor has shown a huge potential to characterize the relative humidity of bulk concrete; however, limited information is available regarding the suitability of embedding MEMS sensor at the steel-concrete interface. In Chapter 4, a novel technique to monitor relative humidity at the steel-concrete interface that can be applied in laboratory-based research is presented. The effects of cementitious materials and moisture content on wireless communication range and transmitted power were examined. Changes in relative humidity for various nominal cover depths, curing age and exposure conditions were also investigated.

The potential of applying the developed technique for in-situ monitoring was evaluated by inspecting the reliability of MEMS sensor. In Chapter 5, MEMS sensors were exposed to 1 year of cyclic wetting and drying, embedded in high and low-strength concrete for 7 months, and directly exposed to 3% wt. NaCl salt solution and 100% RH environment for 3 months. The degradation of sensors was analysed using SEM-BSE and SEM-EDS to study the effects of cementitious material, moisture, and corrosive environments. Changes in relative humidity were compared with unexposed MEMS sensors to study the influence on performance.

Chapter 6 presents a new method to measure the relative humidity of air voids in cement-based materials subjected to wetting-drying. This method is capable of monitoring changes in relative humidity of air voids at the millimeter scale. Results were compared with conventional gravimetric measurements to assess the water absorption and desorption characteristics. Surface morphology and cracks were characterised by stereomicroscope and SEM-BSE as supporting evidence.

Finally, Chapter 7 summarises the main findings from this thesis, and Chapter 8 gives suggestions and recommendations for further work.

Chapter 2 Literature review

This chapter provides a comprehensive review of existing literature relevant to the scope of this thesis. This includes an overview of the electrochemical process of reinforcement corrosion, the influence of macroscopic voids on corrosion initiation, and sensor technologies and imaging techniques with the potential to monitor moisture content at the steel-concrete interface.

2.1 Reinforcement corrosion

Corrosion is an electrochemical process that occurs within reinforced concrete structures. With a difference in electrical potential, electrochemical cells can form at the surface of the steel with anodic and cathodic regions in pore water acting as an electrolyte. At anodic regions, iron oxidizes to produce ferrous irons and reacts with hydroxide ions to form ferrous hydroxide. This can further react with moisture and oxygen to produce rust (Equation 2-1-3). Overtime, corrosion products can accumulate to 2 – 6 times the volume of steel and cause the concrete cover to crack and spall resulting in bond deterioration and a reduction in loadbearing capacity (Marcotte 2001).



Nevertheless, corrosion is significantly inhibited with the formation of a passive film ($\gamma\text{-Fe}_2\text{O}_3$) during the hydration of cement (U. M. Angst et al. 2017; Chen and Orazem 2016). It has an overall thickness of 3 – 15 nm and is capable of decreasing ionic and electronic conduction (Williamson and Isgor 2016). The

passive state can remain uninterrupted at the steel-concrete interface for a long time; nonetheless, the ingress of chlorides and carbonation will eventually break down the passive layer.

Carbonation involves the reaction between gaseous carbon dioxide (CO_2) with cement hydration products such as portlandite ($\text{Ca}(\text{OH})_2$) and calcium silicate hydrate (C-S-H) to form calcium carbonate (CaCO_3). This reduces the pH in the pore solution and the state of passivity at the surface of the steel (Broomfield 2007). The rate of carbonation is critically dependent on the amount of moisture in pores. By controlling the relative humidity of the exposure environment between 50 and 70 %, carbonation was found to reach a maximum rate (Wierig 1984). At lower relative humidity, chemical reactions between calcium-bearing constituents and gaseous CO_2 are limited by a deficiency of moisture (Leemann et al. 2015).

Chlorides in sufficient quantity, 0.5 to 1.0 chloride-to-hydroxyl ion ratio ($[\text{Cl}^-]/[\text{OH}^-]$), can depassivate the steel and initiate corrosion at localized locations (U. M. Angst et al. 2022). This is dependent upon entrapped air voids at the steel-concrete interface and the moisture state of these voids. For example, a loss in the passivity of steel bars was observed near regions of voids with a formation of local anodic areas (Mohammed et al. 2002). High concentrations of Fe and O were discovered in voids less than 500 μm away from corrosion zones (H S Wong et al. 2010). Corrosion pits had developed in close proximity to voids at the steel-concrete interface in 20 year old concrete cores (E. Rossi et al. 2020). However, a number of studies have reported that corrosion rarely coincides with the location of interfacial voids (U. M. Angst et al. 2011; Käthler, Angst, and Elsener 2018). Thus, it was hypothesized that moisture conditions of interfacial voids will probably influence chloride-induced corrosion at the steel-concrete interface (U. M. Angst et al. 2019). Completely saturated interfacial voids containing chlorides may potentially increase the risk of corrosion and buffer the pH during early pitting corrosion. Partially saturated interfacial voids will have a higher abundance of oxygen supply and accelerate the rate of corrosion (U. M. Angst et al. 2019).

Once reinforcing steel progresses from a passive to an active state, the rate of corrosion is mainly affected by the availability of moisture and oxygen. A lack of oxygen and moisture will limit the cathodic reaction and suppress the electrochemical process. Other factors include environmental conditions, cement composition and induced cracks.

2.2 Steel concrete interface

The steel-concrete interface is a transition zone where steel depassivates from passive to an active state and plays an important role in the structural and durability performance of reinforced concrete. However, limited knowledge of local characteristics at the steel-concrete interface has resulted in large uncertainties in durability engineering. This is contributed by the lack of methods to characterize and evaluate the steel-concrete interface, specifically the moisture state. In the following sections, known local characteristics at the steel-concrete interface including the passive film, concrete microstructure and chemistry, and macroscopic interfacial voids will be discussed and presented.

2.2.1 Passive film

The evolution of passive film in alkaline solutions at different pH has been studied by electrochemical techniques. During the formation of the passive film, an increase in open circuit potential (Abd El Haleem et al. 2010; Ghods et al. 2010; Poursaeed and Hansson 2007) and polarization resistance (Sánchez et al. 2007; Volpi et al. 2015) with the time of exposure can be observed. For pH greater than 13, protective attributes are present at approximately 1 day of immersion and 3-5 days in saturated Ca(OH)_2 at pH 12.5. Furthermore, surface analysis has indicated that the passive film consists of an inner Fe^{2+} oxy-hydroxide (1-3 nm) and outer Fe^{3+} hydroxide coat (5-10 nm) with an overall thickness of 3-15 nm (Chen and Orazem 2016; Ghods et al. 2011; A. Rossi, Puddu, and Elsener 2006).

2.2.2 Concrete microstructure and chemistry

At the steel-concrete interface, the initial porosity is relatively high compared to the bulk cement paste. This is resulted by the reduced packing density of cement grains at straight boundaries due to the wall effect. For example, an increase in porosity and accumulation of Ca(OH)_2 was discovered on surfaces of steel with a 40 μm wide porous zone at the underside of horizontal reinforcement (Horne, Richardson, and Brydson 2007a). Microstructure studies conducted by Horne et al. (2007b) revealed an increase in porosity at the aggregate-cement paste interface compared to the bulk cement paste. The difference in porosity can influence the sorption isotherm and degree of saturation at the steel-concrete interface.

2.2.3 Macroscopic voids

During the compaction of fresh concrete, macroscopic interfacial voids can be entrapped under or in close approximation to reinforcing steel bars. These include settlement and bleeding zones, and mechanical damage such as cracks, slip and separation (U. M. Angst et al. 2017). Visual inspection has shown that entrapped air voids are generally larger than 1 mm in diameter with irregular shapes caused by the compaction and contour of surrounding aggregate (U. M. Angst et al. 2017). Bleed water voids have an elongated or crescent shape with a greater contact area at the surface of the steel reinforcement (U. Angst 2011). The absence of calcium-rich hydration products in interfacial voids can significantly reduce the inhibitive effects associated with resistance to a localized pH fall and influence corrosion rate (Glass, Reddy, and Buenfeld 2000). However, there are apparent conflicting evidence and opinions due to an insufficient understanding of these relationships (2.1). It was also hypothesized that interfacial voids can be influenced by other factors such as moisture conditions (U. M. Angst et al. 2019).

2.3 Monitoring techniques

Moisture content in cementitious material and at the steel-concrete interface plays a critical role in the durability and service life of reinforced concrete structures. With a sufficient quantity, carbonation and chloride-induced corrosion can be significantly affected (2.1). However, a fundamental understanding of its properties and effects on corrosion initiation remains inconclusive (Hong S. Wong et al. 2022).

In the following section, existing in-situ and ex-situ methods to monitor the moisture content of the bulk concrete will be examined to establish a suitable technique for characterizing the moisture state of bulk concrete and entrapped air voids at the steel-concrete interface. This includes considerations of scientific principles, sensor components, sensing techniques, embedment methods and applications to provide a better understanding of the accuracy and repeatability to monitor relative humidity between a range of 70 – 90 % . In particular, the ability for quantification within the millimeter range is required to characterize moisture content in macroscopic interfacial voids. Last but not least, the potential for in-situ monitoring on real structures with a reasonable sampling regime and communication schedule is essential for reliable assessment of corrosion risk.

2.3.1 In-situ

Techniques developed for in-situ monitoring are mainly studied with embedded sensors by correlating changes in moisture content with an electrical response. This allows a continuous measurement of the property of interest at a specific location. Before conducting experimental studies, sensors are calibrated in saturated salt solutions with known relative humidity and further evaluated in accordance to reference humidity.

Sensor technologies include:

- Electrical resistance and capacitance
- Fiber optic and Bragg grating sensors
- Microelectromechanical systems (MEMS)
- Microwave and radiofrequency sensors
- Nuclear magnetic resonance sensors

2.3.1.1 Capacitance

Capacitance is a physical quantity used to quantify the dielectric properties of a medium. Thus, a higher moisture content will result in an increase in permittivity of the sensing element and measured capacitance. This changes the resonant frequency of the sensor which can be monitored with an impedance analyzer. During measurement, a voltage will be applied to an electrode plate and changes in permittivity (ϵ) of the sensing element with a predetermined area (A) and distance (d) can be determined (Equation 2-4). The sensor is typically constructed with two electrode plates and a hygroscopic polymer or metal oxide film acting as a dielectric medium (Figure 2-1).

$$C = \frac{A}{d} \epsilon \quad 2-4$$

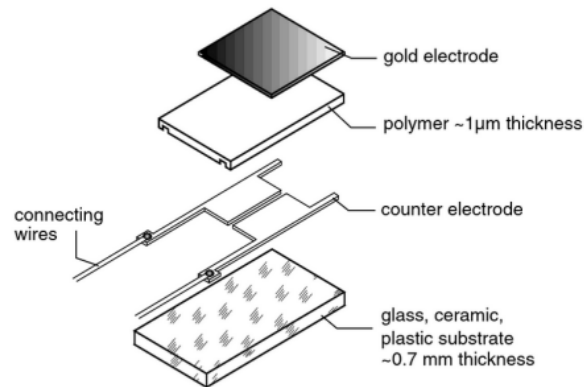


Figure 2-1 A typical structure of a capacitive humidity sensor (Raunima et al. 2019).

To monitor moisture content in cementitious and porous material, capacitance sensors are typically encased in protective coatings and porous filters to prevent shorting of circuit components and condensation. A typical example is the application of paraffin wax on an inductor-capacitor resonant circuit (LC) sensor with inner and outer lengths of 2.5 and 4cm to monitor the reduction in moisture content of sand and mortar samples (You et al. 2008). Although the sensor was able to measure a lower moisture content for materials with a lower porosity with an average resonant frequency of 21.77, 20.74 and 19.95 MHz for sand samples with the respective particle sizes of 2.36, 0.6 and 0.15 mm.

Condensation on the sensor surface resulted in a higher signal-to-noise ratio and a reduction in resonant quality. A similar observation was made by Raunima et al. (2019) with inconsistent results measured at relative humidity above 90 %. Therefore, when used to monitor moisture content at the steel-concrete interface, condensation can cause inaccurate measurements and dimensions are typically above the millimeter range which is not feasible for monitoring moisture content in macroscopic voids. Moreover, the application of protective coatings and filter elements can further impact the overall measured accuracy and reliability.

2.3.1.2 Resistance

Resistance is determined based on the amount of charged ions in cementitious materials. The presence of moisture will increase the electronic and electrolytic current flow and generate a lower resistance value. To measure resistance, it is recommended to apply an alternating current (AC) with a frequency between 50 and 1000 Hz (Polder 2001), and higher frequencies of 100Hz to 10kHz are recommended for low

temperature and high humidity environments. A theoretical relationship between current (I) and resistance (ρ) can be determined based on Ohm's law (Equation 2-5). To obtain measurements, each electrode pair is attached to an auto ranging logger with a cable terminal (Figure 2-2 a). In addition, electrodes are placed at a predetermined distance to obtain meaningful information regarding moisture transport and ionic movement (Figure 2-2 b).

$$\rho = \frac{\Delta V}{I}$$

2-5

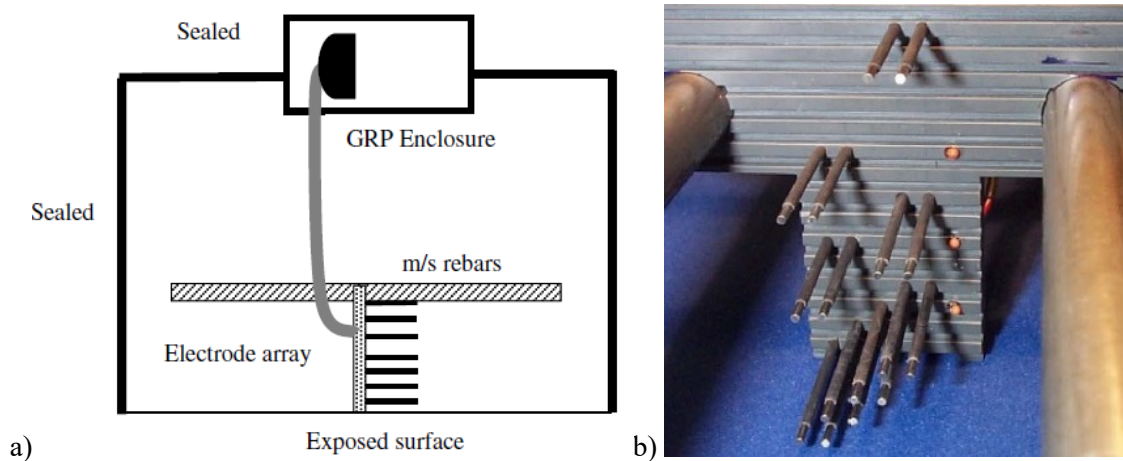


Figure 2-2 Illustration of the multi electrode array with a) a schematic design and b) distribution at different cover depths (McCarter et al. 2012).

Moisture content in close proximity to the steel has been characterized by resistivity sensors. Examples include examining the ingress of moisture with a multi-electrode array exposed to marine conditions (McCarter et al. 2012). However, the proposed method requires electrodes to be electrically isolated from the steel reinforcement with a stainless steel pin sleeve and applying an alternating current voltage of 1.0 V and fixed frequency of 1 kHz to minimize polarization effects. Zaccardi et al. (2009) examined changes in resistance by exposing cylindrical mortar specimens to various temperatures and relative humidity. Slight deviations in resistance measurements were observed due to a shift of sensor position from different compaction methods. This sensor technology can potentially monitor the relative humidity of bulk concrete and macroscopic voids at the steel-concrete interface; however, alternative embedment methods are required to improve the reliability of measurements.

2.3.1.3 Fiber Optic and Bragg grating

Fiber Bragg grating (FBG) functions by integrating an optical filter that reflects a range of incident light signals with a peak wavelength called the Bragg wavelength. The presence of moisture expands a moisture sensitive polymer that induces strain in the FBG and changes the Bragg wavelength (Chai et al. 2018). This can be monitored with an optical spectrum analyser or other wavelength-based interrogation methods (Figure 2-3). These attributes have been implemented to study relative humidity changes during desorption (X. Lu et al. 2021), ingress of moisture (Yeo, Cox, et al. 2006) and moisture absorption (Yeo, Eckstein, et al. 2006) in cementitious materials. Feasibility studies conducted by Yeo et al. (2006), demonstrated that FBG sensors coated with polyimide can detect a substantial amount of moisture retained in 0.5 w/c samples compared to 0.7 w/c samples. However, changes in response was observed as a result of condensation. Similar observations were made by Lu et al. (2021), with results indicating the impact of the scattering coefficient and the attenuation of fiber on measurements. The diameter of fiber Bragg grating (FBG) sensors is typically within the millimeter range and can potentially be embedded at the steel-concrete interface to measure changes in relative humidity of bulk concrete and macroscopic voids. However, sensors must be calibrated to compensate for scattering effects and attenuation.

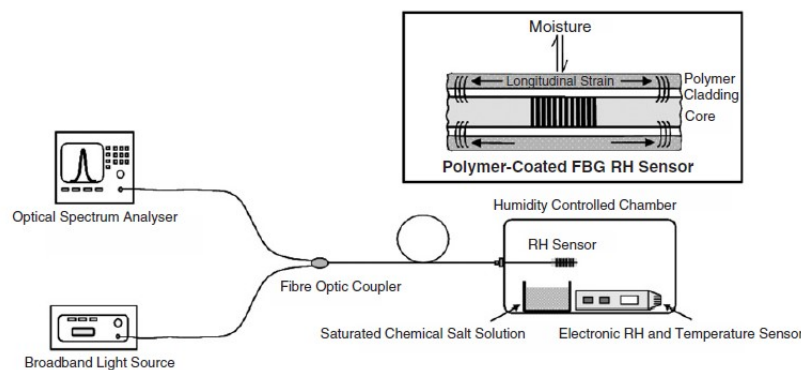


Figure 2-3 Schematic design of experimental setup to obtain relative humidity measurements from polymer coated FBG sensor (Yeo, Eckstein, et al. 2006).

2.3.1.4 Microelectromechanical systems

Microelectromechanical systems (MEMS) sensors function by coating an expanding nano-water vapour polymer film on cantilever beams. An increase in moisture content induces stress and strains that are monitored with embedded resistors (Figure 2-4). The potential of monitoring moisture content in concrete

was studied by packaging MEMS sensors in a stainless steel enclosure with dimensions of 5mm diameter and 3mm thickness (Norris, Saafi, and Romine 2008). Results demonstrated that MEMS sensors can detect a 60 % difference in relative humidity for specimens with a 50 % reduction in sample size. Other examples include examining moisture content in early age concrete by depositing and bonding a sensitive polymer (PolyViol G 2810) on MEMS sensors (Saafi and Romine 2005). A higher humidity response was observed for samples with a higher water to cement ratio and measurements were unaffected after subjecting concrete cylinders to compression. This sensor technology has shown a higher potential to monitor moisture content at the steel-concrete interface with reliable measurements when subjected to mechanical load and obtainable measurements after 150 days of embedment. It is also feasible to monitor moisture content in macroscopic voids due to the small sensor size.

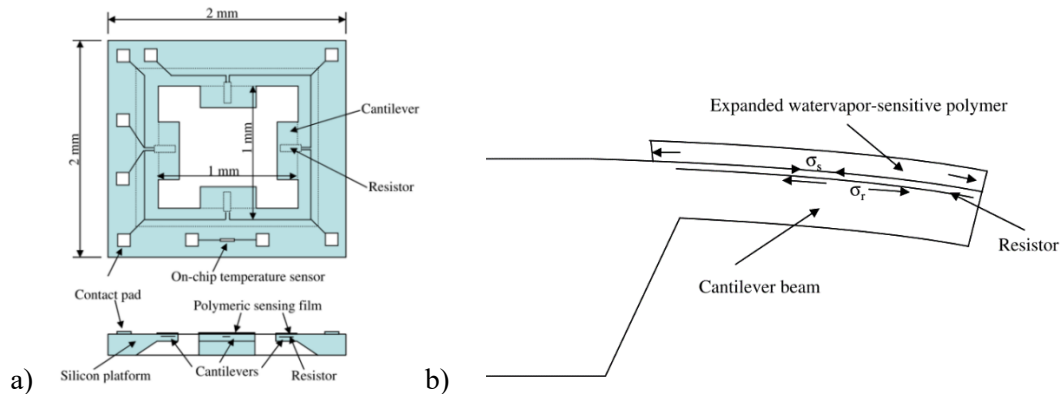


Figure 2-4 MEMS microcantilever sensor with a) a schematic design and b) functionality of the expanded water vapour sensitive polymer (Norris, Saafi, and Romine 2008).

2.3.1.5 Microwave and Radio Frequency

Electromagnetic waves and dielectric properties have been extensively studied for material and structural assessment. The high polar characteristics of water molecules will result in a higher relative permittivity and lower oscillation frequency that can be characterized and monitored with a frequency meter. This enables direct relationships between applied oscillation frequency and moisture content to be drawn by calibrating sensors in a climatic test cabinet with a scale to monitor changes in mass (Figure 2-5). Sokoll et al. (2014) developed a compact and inexpensive microwave sensor consisting of a moisture-sensitive material, resonator, protective foil and transmission line. Measurements indicate that the developed sensor corresponded well to moisture changes when subjecting concrete specimens to wetting and drying cycles.

Nevertheless, fluctuations in sensitivity can occur after long-term localized moisture monitoring. To minimize this effect, filler materials made of gypsum and mortar were cast to encapsulate exposed wires and minimize gaps between conductors and the measured interface (Malan et al. 2004). Findings from this study showed that mortar was more chemically compatible with cementitious materials and can be used to monitor changes in moisture content. Nonetheless, fluctuations in sensitivity can potentially pose problems in the accuracy of measurements that needs to be addressed before embedding this sensor technology at the steel-concrete interface for monitoring moisture content.

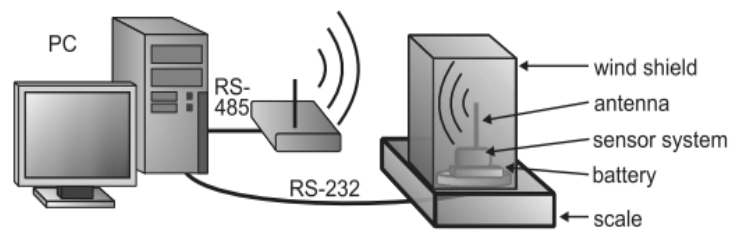


Figure 2-5 Schematic of calibration system to obtain direct relationships between applied oscillation frequency and moisture content (Sokoll and Jacob 2014).

2.3.1.6 Nuclear magnetic resonance

With the emission of an electromagnetic field, an increase in moisture content will prolong the transverse relaxation time of protons with an irreversible decay of phase relationships between individual spins; hence, such attributes can be extrapolated to a signal amplitude to characterize moisture transport (Nestle, Galvosas, and Karger 2007). This includes calibrating sensors to the Larmor frequency by adjusting the magnetic field strength to 0.24 T with metallic objects (Figure 2-6). For cementitious materials, nuclear magnetic resonance (NMR) has been extensively adopted to study hydration (Muller et al. 2015), moisture distribution during desorption (P. F. de J. Cano-Barrita et al. 2004) and capillary water absorption (Fischer, Haerdtl, and McDonald 2015). Since then, miniaturized NMR sensors were developed and embedded in mortar to monitor moisture changes during hydration and drying (P. F. de J. Cano-Barrita et al. 2009). The sensor consists of a radio frequency coil tuned to the Larmor frequency (≈ 10.2 MHz) with capacitors etched on a printed circuit board placed above two neodymium (NdFeB) disk magnets encased in waterproof epoxy resin. Measurements demonstrated that the developed sensor was

sensitive to moisture changes and able to detect a reduction in signal amplitude after subjecting specimens to 60 °C; however, measurements have a lower absolute signal-to-noise ratio and limited depth range. Thus, this sensor technology will not provide sufficient spatial resolution to characterize moisture content in macroscopic voids.

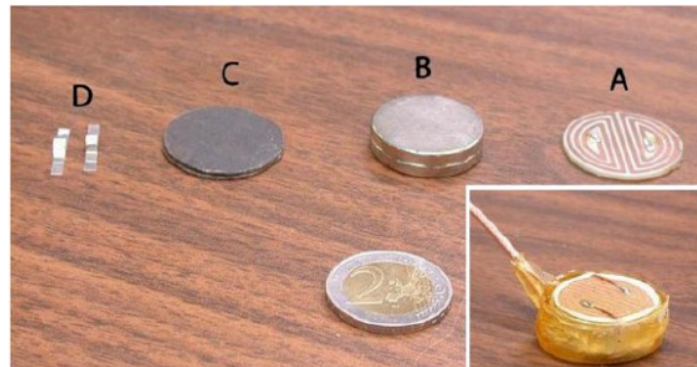


Figure 2-6 Internal components of NMR sensor (P. F. de J. Cano-Barrita et al. 2009).

2.3.1.7 Piezoelectric sensors

During embedment, digital signals are generated by a waveform generator and transformed into stress wave signals by piezoelectric actuators based on the inverse piezoelectric effect. Moisture will induce changes in the harmonic amplitude and can be examined by a wavelet packet energy analysis. This is because incremental changes of water depth and water content will increase the elastic modulus and result in a decrease in the waveform amplitude. Typically the internal components of the sensor consists of a piezoelectric material integrated between two marble blocks attached to lead wires (Figure 2-7). Early studies have shown piezoelectric sensors constructed from lead zirconate titanate patches were able to monitor water seepage in concrete; however, internal damage induced by mechanical loading and aggregate particles can influence the accuracy of measurements (Liu et al. 2013). Since then, further investigations on early age concrete hydration (Kong et al. 2013) and internal relative humidity were conducted (H. Zhang, Li, and Kang 2022). Findings from these studies showed that piezoelectric sensors have a good sensitivity to moisture, but fluctuations in ambient temperature and properties of coating material will affect measured signal amplitude. Therefore, the influence of internal damage induced by loading, temperature, coating materials and cementitious materials on signal response needs to be further investigated before this sensor technology can be embedded at the steel-concrete interface to monitor

moisture content. Furthermore, it is unfeasible to monitor moisture content in macroscopic voids with this sensor technology due to the large sensor size.

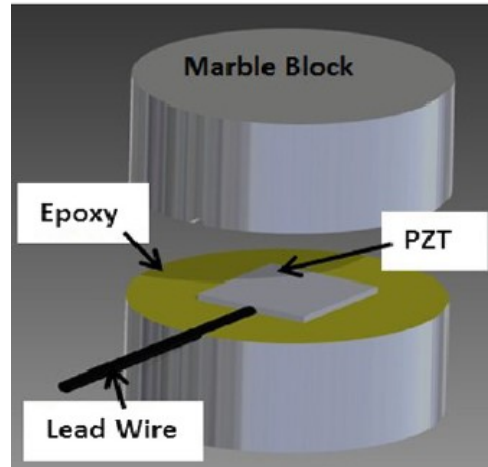


Figure 2-7 Internal components of the piezoelectric sensor (Kong et al. 2013)

2.3.2 Ex-situ

Ex-situ monitoring is an external visualization method that examines moisture transport processes in cementitious material without interfering with the overall integrity and composition of a specimen. These include unsaturated moisture flow (Dalton et al. 2022; M. Zhang 2017), and the movement of the water front (Lal et al. 2014; Oesch et al. 2019; Zhao et al. 2021).

Visualization techniques include:

- X-ray tomography
- Neutron imaging
- Nuclear magnetic resonance imaging

2.3.2.1 X-ray tomography

X-ray tomography is a technique that involves the projection of X-ray onto samples to examine moisture content (Figure 2-8). Captured images are further processed into a volumetric dataset with the Feldkamp back-projection algorithm and imported into software tools for quantitative analysis (Feldkamp, Davis, and Kress 1984). The analysis procedure involves segmenting tomography images to distinguish air, cement and voids, and normalizing CT number with a unified CT number scale to obtain a water content profile. Examples include the study of movement and 3D spatial distribution of moisture in concrete beams (Oesch et al. 2019) and capillary absorption in cementitious material (Hong et al. 2020). Although X-ray tomography provides the ability to measure the height of water absorption front and water content gradient from three dimensional images. Artefacts (cupping and dark streaks) induced during the projection of X-ray with higher CT number at outer regions of specimens creates difficulties for quantitative analysis. This can be potentially improved by calibrating computed attenuation values from a polychromatic to a monochromatic characteristic (Krumm, Kasperl, and Franz 2008). However, artefacts along the edges of aggregates and pores with overestimated moisture content gradients still persist. Thus, this approach can potentially be applied to quantify the temporal and spatial distribution of moisture content close to the steel-concrete interface but further improvements are required to reduce artefacts induced by this technique.

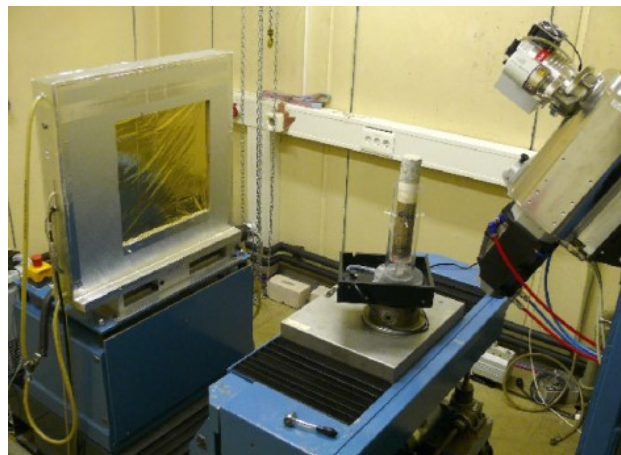


Figure 2-8 Overview of experimental setup to examine water transport in concrete (Oesch et al. 2019).

2.3.2.2 Neutron imaging

Neutron imaging is a technique that has been widely applied to investigate water-related durability issues in cementitious materials (Toropovs et al. 2015; Trtik et al. 2011; P. Zhang et al. 2010). This is because neutrons are attenuated by hydrogen present in moisture and are less sensitive to other elements found in cement-based materials such as Si, Ca, Al, Fe, S, O, Na and K. The system consists of a detector, neutron beam and rotating table with acquired radiograms normalized to account for background noise, inhomogeneity and electronic noise (Figure 2-9). For example, Duati et al. (2018) examined moisture migration in concrete at elevated temperatures and detected slower desorption rates for specimens with smaller maximum aggregate sizes. Lal et al. (2014) investigated the water uptake in porous asphalt by combining neutron and microtomography imaging. They observed a rise in saturated flow between binders-aggregate interfaces and a slower uptake of moisture between lateral boundaries of pores. However, this technique has been rarely explored to characterize moisture content at the steel-concrete interface. Zhang et al. (2010) investigated water penetration in cracked steel-reinforced mortar and discovered an increase in water content through a load induced crack at the steel-mortar interface. Methods proposed from these studies indicate that neutron imaging can be a promising technique to monitor the moisture state of bulk concrete and macroscopic voids at the steel-concrete interface; nonetheless, the acquisition time of 2D and 3D tomographs has to be carefully considered.

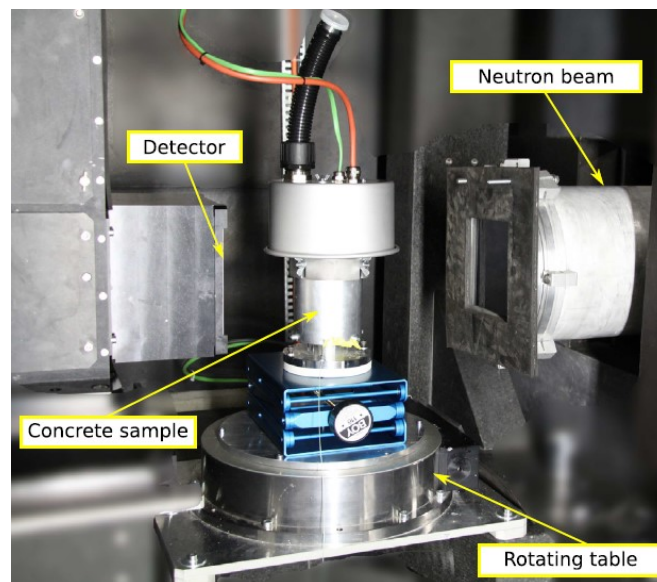


Figure 2-9 Experimental setup to analyze moisture migration in concrete at high temperatures (Duati et al. 2018)

2.3.2.3 Nuclear magnetic resonance imaging

^1H nuclear magnetic resonance imaging provides the ability to quantify moisture in cementitious materials by correlating gravimetric measurements with nuclear magnetic resonance signal intensity (Gajewicz 2014). Typically, the imaging system consists of an elliptical radio frequency coil to generate electromagnetic fields and a Faraday cage to improve the signal-noise ratio (Figure 2-10). Changes in nuclear magnetic resonance signal intensity is dependent on the relaxation time of the hydrogen nuclei spins and dead time between the excitation pulse and initial signal acquisition time of the radio frequency coil. These attributes have been evaluated to study the absorption of moisture in mortar prisms with different moisture content (Gramm and Beddoe 2010). They observed an increase in NMR signal intensity in proportion to water content at above 80 % RH. However, NMR scans were insensitive to moisture and were not able to detect moisture in pores below 80 % RH. Hence, this approach can potentially provide the spatial distribution of moisture content at the steel-concrete interface; however, the sensitivity to moisture must be further improved and the influence of reinforcing steel on magnetic fields must be considered. In addition, the spatial resolution of this technique is insufficient to measure moisture content in macroscopic voids.

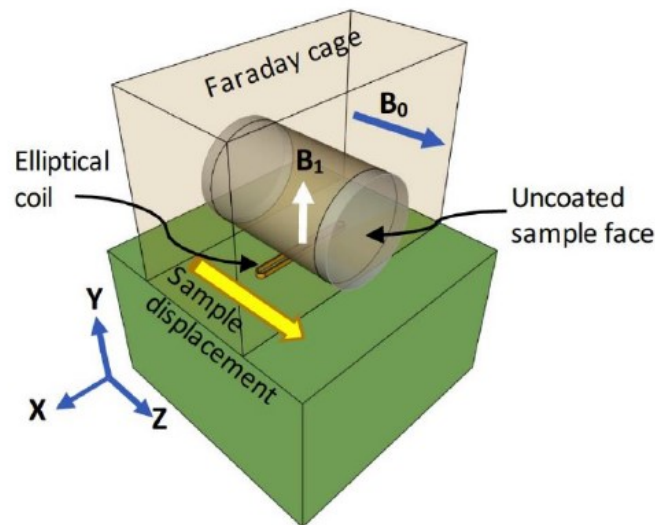


Figure 2-10 Experimental setup for ^1H nuclear magnetic resonance imaging to examine moisture content (P. F. Cano-Barrita and Díaz-Díaz 2021)

2.4 Discussion

Characterizing moisture content at the steel-concrete interface is particularly challenging because of its complex physical and chemical nature. This includes changes in properties at the steel surface from various exposure and environmental conditions, e.g., hydration, mechanical load, ingress of moisture and diurnal air temperature. Furthermore, the metallurgy and geometry of reinforcing steel can significantly affect the chemical composition and microstructure of the bulk concrete at the steel surface (2.2.2).

From the reviewed monitoring technologies, ex-situ visualization methods have shown the potential to quantify the spatial and temporal distribution of moisture content at the steel-concrete interface. However, each technique has drawbacks. Neutron imaging can examine the migration of moisture between binders-aggregate interfaces and moisture content at the steel-mortar interface. Nonetheless, 3D tomographs can result in acquisition times of at least 13 minutes per radiography. X-ray tomography was capable of monitoring the spatial distribution of moisture and height of water absorption front; however, the low X-ray attenuation of water and artefacts can create difficulties for quantitative analysis. Nuclear magnetic resonance can detect changes in moisture content from the excitation of the hydrogen nucleus. But results can be influenced by hydrogen-bearing products in hydrated cement paste such as Calcium-Silicate-Hydrate (C-S-H) gel and Calcium-Hydroxide (CH).

On the other hand, in-situ monitoring techniques can provide a temporal distribution of moisture content at the steel-concrete interface with direct measurements. Nevertheless, condensation and changes in temperature can affect the reliability and accuracy of capacitance, resistance and piezoelectric sensors. Additionally, studies have indicated sensitivity issues for fiber Optic and Bragg grating and microwave and radio frequency sensors. Therefore, MEMS sensors have a higher potential to monitor the moisture content of the bulk concrete and macroscopic interfacial voids at the steel-concrete interface for laboratory-based research. This is based on the criteria that reliable measurements can be obtained including the ability to monitor saturated and partially saturated conditions which are particularly important for durability engineering. Moreover, MEMS sensors can be paired with wireless technology and permanently embedded in concrete for long-term monitoring. Thus, the potential of applying MEMS sensors for in-situ monitoring on real structures can be further examined.

Regardless, both ex-situ and in-situ techniques are mostly applied in laboratory-based research and the feasibility of developing techniques for in-situ monitoring on real structures is rarely explored. This can be attributed to the challenges and difficulties of creating appropriate methods for sensor embedment. By

placing sensors at the steel-concrete interface, the high conductivity of steel and the accumulation of aggregates can impact the accuracy of measurements. In addition, exposure to alkaline, humid and corrosive environments can degrade electronic and sensor components. This includes the addition of additives to enhance the properties of concrete to achieve specified requirements. Therefore, suitable embedment methods must be investigated to enhance the long-term performance and durability of sensors. The influence of additives is not examined in this study to provide a better understanding of the impact of Portland cement and cement paste pore solution on durability and performance of sensors.

Table 2-1 Overview of technologies and their potential for monitoring moisture at the steel-concrete interface.

Sensor technology	Repeatability (%)	Accuracy (%)	Range/ Resolution	Stability	Potential	Enhancements
1. Resistance	12	7.2	40-100 RH %	Highly influenced by temperature changes	Medium	Specimen geometry is examined numerically and experimentally to improve resistivity measurements
2. Capacitance	10	10	33-97 RH %	Fluctuates at relative humidity greater than 90 %	Medium	Application nano particles such as zinc oxide coating can improve sensitivity at low moisture content between 7-25 %
3. Fiber Optic and Bragg grating	0.03	4	16-94 RH %	Oxygen-hydrogen vibrational absorption can cause attenuation at certain wavelengths	Medium	Sensing fibers with a poly(methyl methacrylate) core and optical time domain reflectometer have been explored to

						improve weak scattering effects
4. Microelectro-mechanical systems	0.003	0.03	6-90 RH %	Sensor output was not affected after applying compressive loads of 35 MPa	High	AlN, CrN and CrTiN protective coatings have been applied on MEMS to enhance mechanical and corrosion stability
5. Microwave and radio frequency	0.117	10	10-90 RH %	Measurements were within the uncertainty margin (10 %) of the system after embedding in cement and concrete	Medium	Dielectric materials with a hysteresis lower than 7 % RH are being investigated
6. Nuclear magnetic resonance	1	5	5.9 μm	A reduction in sensor length/diameter ratio of 34 % will result an increase in echo time of 68%	Medium	The application of micro-fabricated micro-coils can improve sensitivity and shorter echo times of 10 μs
7. Piezoelectric sensors	3	10	30-100 RH %	During the hardening of cement paste, the signal responses from the time and frequency domain were chaotic	Medium	The influence of epoxy resin properties, water to cement ratio and ambient temperature on signal amplitude is being investigated

8. X-ray tomography	7.8	3	0-12 vol. %	Distortions can be caused by sample expansion during water absorption. Thus, causing artefacts at interfaces of pores and aggregates	Low	XCT is compared with NCT and 3D-NMR to provide better calibration and validation processes
9. Neutron Imaging	-	1.8	5-50 vol. %	Evaporation of water from samples during water absorption can significantly increase the attenuation coefficient	Low	Experiments with improved boundary conditions are being studied to provide a better analysis of water accumulation
10. Nuclear magnetic resonance imaging	3	5	0-30 vol. %	Low sensitivity of NMR signals at moisture content below 20 % which contributes to noise	Low	Three-magnet array with elliptical radio frequency coil is being evaluated to improve spatial resolution

2.5 Impact on reliability and performance of embedded sensors

As discussed in the previous section, MEMS sensors were found to have a higher potential for monitoring moisture content at the steel-concrete interface. However, improper embedment methods and techniques can affect the performance and functionality of sensor components. For example, Strangfeld et al. (2019) embedded RFID-based sensors in concrete to examine the functionality and robustness. They observed sensors operating at the ultra-high frequency range (868 MHz) were not functional after 156 days while

sensors operating at the high-frequency range (13.56 MHz) provided constant measurements for 1016 days. It was speculated that sensor components had deteriorated but findings were inconclusive. Prior to developing a suitable and reliable method to monitor moisture content at the steel-concrete interface known parameters that impact wireless communication, accuracy and durability will be presented and discussed in the following sections.

2.5.1 Wireless communication

Most wireless communication devices transmit and receive measurements by electromagnetic waves. During the propagation of electromagnetic waves, an increase in moisture content in pores and aggregates can change the permittivity of materials, attenuate the radio frequency and reduce the wavelength of incident electromagnetic waves (Asp et al. 2019; Bjorninen et al. 2013; Zemmour et al. 2015; Zemmour, Baudoin, and Diet 2016, 2017). The Nicolson-Ross-Weir method is commonly used to identify the permittivity and permeability of homogenic and isotropic materials with known parameters such as permittivity of vacuum (ϵ_0), relative permittivity of material (ϵ_r), material dielectric constant (ϵ_r') and imaginary dielectric loss factor ($j\epsilon_r''$) shown in Equation 2-6.

$$\epsilon = \epsilon_r \epsilon_0 = (\epsilon_r' - j\epsilon_r'') \epsilon_0 \quad 2-6$$

For example, the influence of water to cement ratio on radio frequency attenuations and permittivity was examined with a pair of spot-focusing reflector antennas (Asp et al. 2019). Measurements obtained from this study demonstrated that material dielectric constant and attenuation were not affected by changes in water to cement ratio and attenuation was significantly reduced at lower frequencies. These findings are in agreement with the current literature that presents attenuation increasing in concrete at higher frequencies (Figure 2-11). At frequencies below 100 MHz, attenuation is similar to free space at 10 dB/m; therefore in many instances, HF radio antennas have been recommended to be encapsulated in concrete (Bilgutay et al. 2001). For frequencies above 1 GHz, a reduction in received power of 20 % is approximated for a concrete cover of 80 mm (Muqaibel et al. 2003).

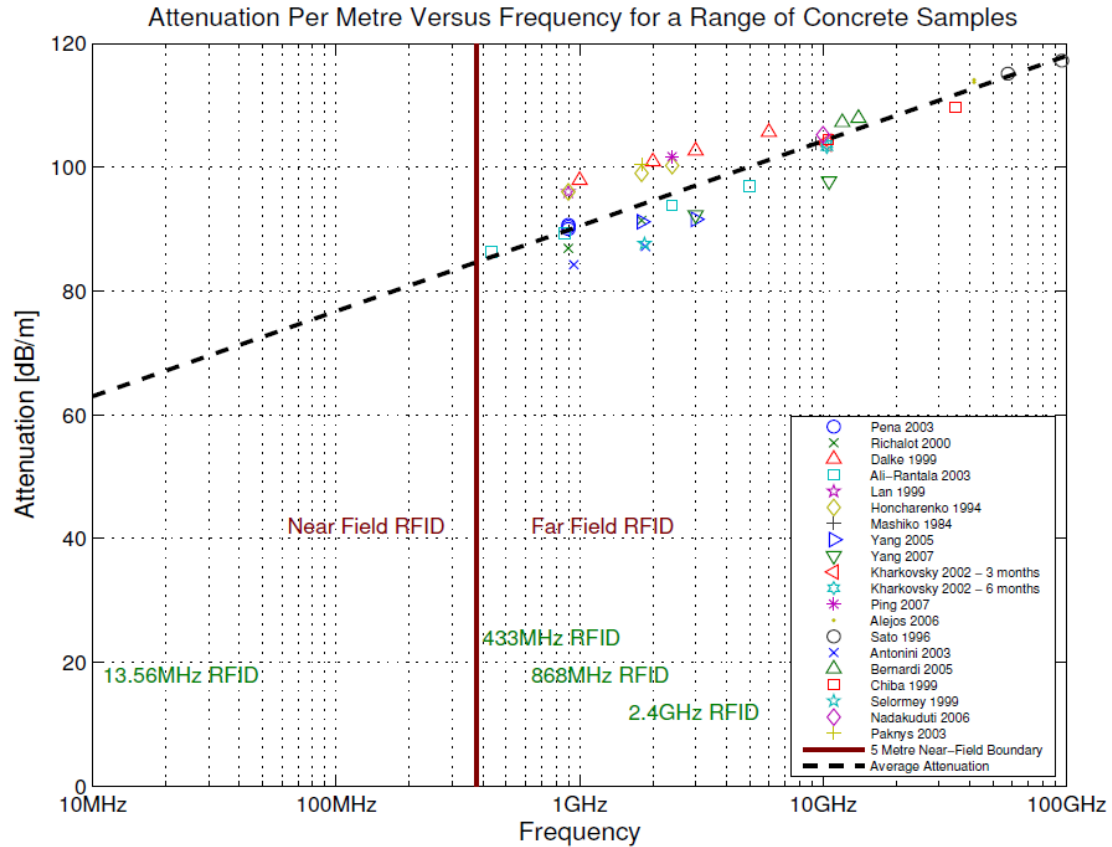


Figure 2-11 Radio attenuation per metre in concrete (Daly, Melia, and Baldwin 2010)

2.5.2 Accuracy

At high relative humidity, water molecules can condense on the surfaces of sensors and induce large capillary forces impacting the mechanical properties of solid contacts and accuracy (Crassous, Ciccotti, and Charlaix 2011). This causes issues related to stiction where interatomic and intermolecular surface forces including electrostatic, van der Waals and inertia can affect measurements and cause failures (Birleanu et al. 2017; Raunima, Tuominen, and Vinha 2019; Zaghloul et al. 2011). Particularly, it is a major concern for micro-devices due to the amplification of surface effects at the nanoscale. After exposing 300 nm thick silicon nitride (SiN_x) film to 1 - 80 % relative humidity environments an increase in the adhesive force of 500 nN was observed with an atomic force microscope (Zaghloul et al. 2011). Patton et al. (1999) discovered a reduction in performance and high stiction forces by placing MEMS lateral output motor in relative humidity environments above 70% RH. A rise in pull of forces from 20 to

22 nN on aluminium bridges and silicon substrates was observed by increasing the relative humidity of the surrounding environment from 40 to 70% RH (Birleanu et al. 2017). Lower fatigue strength was measured for 3µm thick silicon thin films exposed to relative humidity at 85-90% compared to laboratory conditions at 50-65% (Yamaji et al. 2007).

2.5.3 Durability

Environments with high alkalinity, moisture and chloride content can oxidize and degrade electronic components impacting the long-term durability and function of sensors (Broas et al. 2015; Donahoe et al. 2006; Guo, Niu, and He 2015; Hon et al. 2008; Miller et al. 2006; Nahar 2000; Raman et al. 2011; Seo et al. 2019; Strangfeld, Johann, and Bartholmai 2019; Surendar et al. 2018). For example, exposure to corrosive acids can form oxides and alter the overall surface morphology (Hon et al. 2008; Kahn et al. 2005). High temperatures and relative humidity can result in electrochemical migration and reduce the sensitivity of multilayer ceramic capacitors (Raman et al. 2011). Donahoe et al. (2006) studied the ageing mechanisms of multilayer ceramic capacitors made of silver palladium and nickel with copper electrodes. They discovered that temperature and moisture can impact measured capacitance values after 5000 hours of exposure (Figure 2-12).

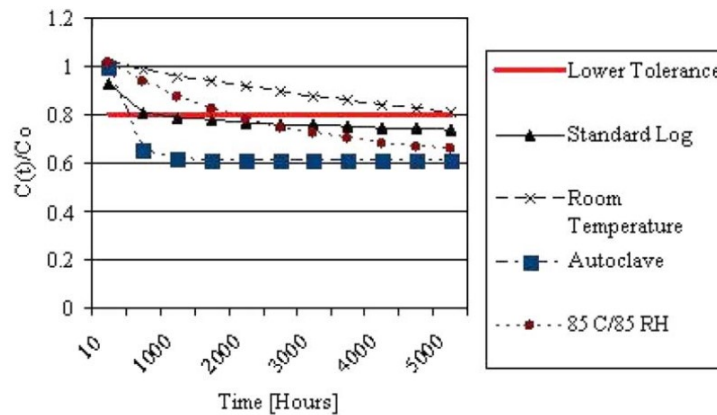


Figure 2-12 Changes in capacitance over time for capacitors exposed to different environmental conditions (Donahoe et al. 2006).

Strangfeld et al. (2019) examined the long-term stability of radio frequency identification (RFID) humidity sensors in cementitious environments. The embedded sensors consist of an analogue humidity sensor (Honeywell HIH-5030) connected to an RFID integrated circuit operating at the high frequency (HF) range (13.56 MHz) and ultra-high frequency (UHF) range (868 MHz). HF RFID sensors were encased in polyvinyl chloride (PVC) casing with the circuit board encapsulated in epoxy resin and UHF RFID sensors were encased in polyvinyl chloride (PVC) casing without epoxy resin (Figure 2-13). Subsequently, sensors were embedded in cementitious screed and extracted after 3 years. They observed signs of discolouration and jagged surfaces on epoxy resin and no signs of degradation were observed for sensors without epoxy resin. (Figure 2-14). Findings from this study also indicated a slight degradation of the capacitive humidity sensor; however, based on visual examination it was uncertain if the polymer membrane has aged (Figure 2-15 a).

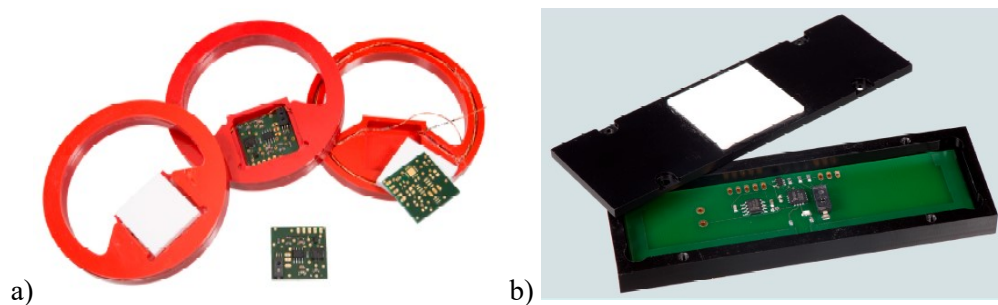


Figure 2-13 Images of embedded a) HF RFID sensors and b) UHF RFID sensors (Strangfeld, Johann, and Bartholmai 2019).

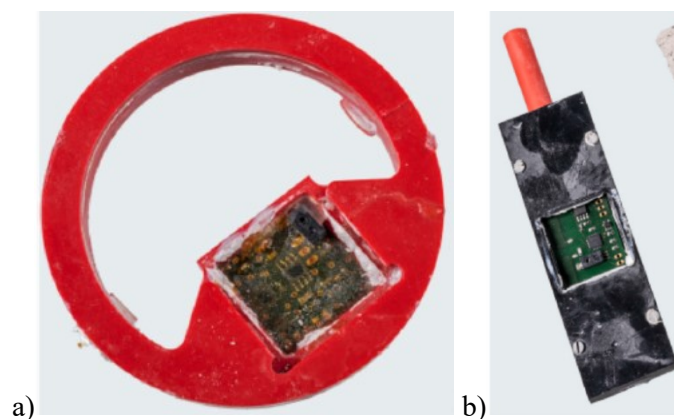


Figure 2-14 Photographs of sensors a) with epoxy resin and b) without epoxy resin (Strangfeld, Johann, and Bartholmai 2019).

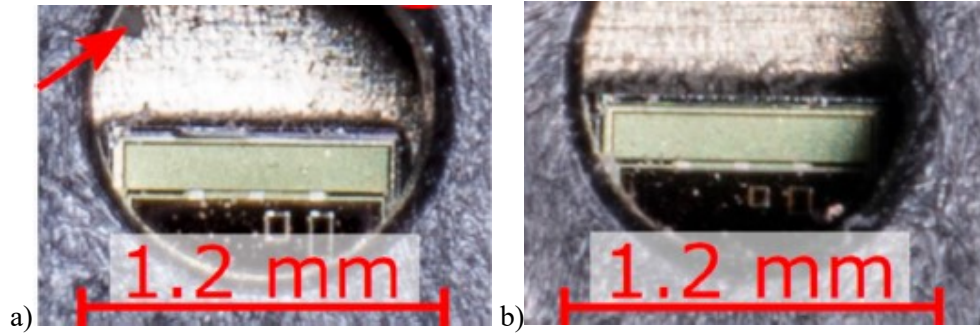


Figure 2-15 Photographs of capacitive humidity sensors a) with epoxy resin and b) without epoxy resin (Strangfeld, Johann, and Bartholmai 2019).

Broas et al (2015) evaluated the degradation of silicon-based thin films with nanoindentation. The MEMS microphone adopted for this study was based on a parallel capacitor membrane consisting of two conducting polycrystalline silicon thin film membranes, a backplate and a diaphragm, with a thickness of 400 nm. Changes in capacitance is detected due to a separation distance between the membranes with an overview of the MEMS package and element shown in Figure 2-16. The assembled MEMS microphones were exposed to a mixed flowing gas (MFG) test for 50 and 90 days in an environmental test chamber (Weiss WK11 600/40) with impurity gases of Cl_2 , H_2S , NO_2 and SO_2 , and parameters are shown in Table 2-2. After exposure, nanoindentation was conducted with a nanomechanical testing instrument (Hysitron TriboIndenter) to induce fracture points in the backplates. Measurements showed a decrease in fracture force for MEMS exposed to impurity gases compared to the unexposed reference sample (Figure 2-17).

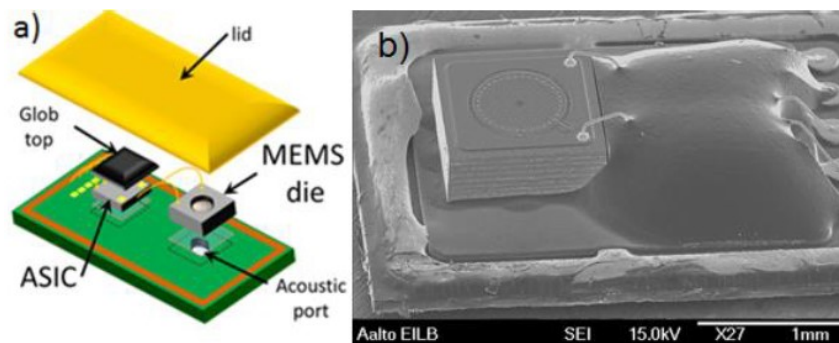


Figure 2-16 Overview of MEMS microphone with a) schematic and b) SEM image of package structure (Broas et al. 2015).

Table 2-2 Mixed flowing gas (MFG) test parameters (Broas et al. 2015).

MFG Test Parameters	Calculated values	GR-63-CORE
Temperature	30°C	30°C
Humidity	70% R.H.	70 % R.H.
Cl ₂	55 ppb	20 ppb
H ₂ S	350 ppb	100 ppb
NO ₂	350 ppb	200 ppb
SO ₂	350 ppb	200 ppb
Air turnover rate	8 times / h	5–50 times /h

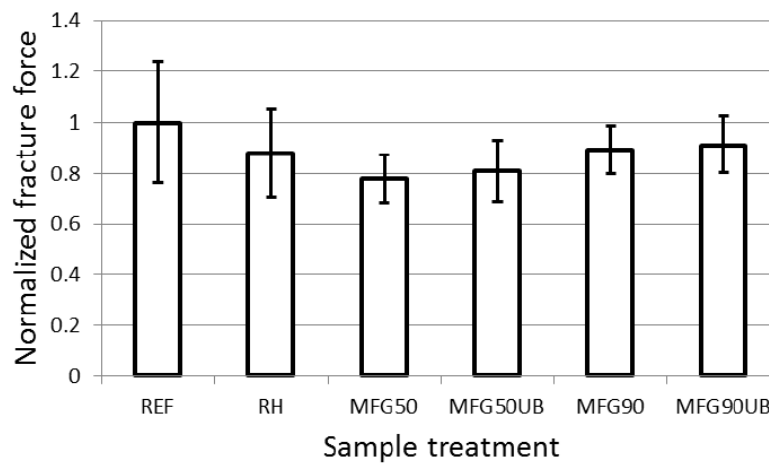


Figure 2-17 Normalized fracture force on MEMS microphone after the exposure to mixed flowing gas test (Broas et al. 2015).

2.5.4 Enhancements

Efforts have been made in recent years to improve the performance and reliability of MEMS sensors. This is achieved by implementing heating elements, alternative sensing materials and suitable packaging techniques to reduce the effects of stiction, corrosion and hysteresis. Dai et al. (2007) integrated a micro heater into a nanowire tungsten trioxide humidity sensor and discovered that sensitivity was approximately 4.5mV/% at 60°C and 4.8mV/% at 75°C with minimal signal drift. Brazzle et al. (2003) substituted platinum alloy as a MEMS flexure material and discovered a mechanical hysteresis of less than 0.0034 degrees. Choudhary et al. (2011) added AlN, CrN and CrTiN protective coating on NiTi/PZT thin films to enhance the mechanical and corrosion stability. Results showed that CrTiN coated

NiTi/PZT/TiO_x films exhibited the lowest corrosion rate and best corrosion resistance when exposed to 1 M NaCl solution. Pederson et al. (2004) implemented an O-ring clamping technique on MEMS piezoresistive pressure sensor and observed an offset of less than 0.4% of the maximum output after 1 million differential pressure cycles.

2.6 Chapter Summary

Existing studies for monitoring moisture content in cementitious materials were reviewed in this Chapter. It is evident that there is a lack of suitable embedment and non-destructive techniques to monitor the moisture content at the steel-concrete interface. Current methods provide no clear indications of appropriate procedures to embed sensor technologies in cementitious materials. Limited studies have investigated the effects of cementitious materials on the durability and reliability of sensor components. There is also a lack of research on evaluating the moisture state of concrete and entrapped air voids at the surface of the steel. Understanding these effects can provide a more reliable assessment of corrosion risk and help to reduce uncertainties in durability engineering. This thesis aims to contribute toward these issues.

Chapter 3 Methodology

This chapter presents materials and experimental procedures adopted in this thesis. Details of materials, sample preparation and instrumentation are presented.

3.1 Materials and mix proportion

To ensure the suitability of RFID MEMS sensors to monitor relative humidity in structural and non-structural elements, specimens with the desired design mix composition were casted (3.1.2). The specific mix proportions were adjusted and determined based on known properties of Portland Cement, Thames Valley sand and gravel (3.1.1). Furthermore, samples were conditioned to a standard moisture state to characterize water absorption and desorption attributes (3.1.3).

3.1.1 Materials

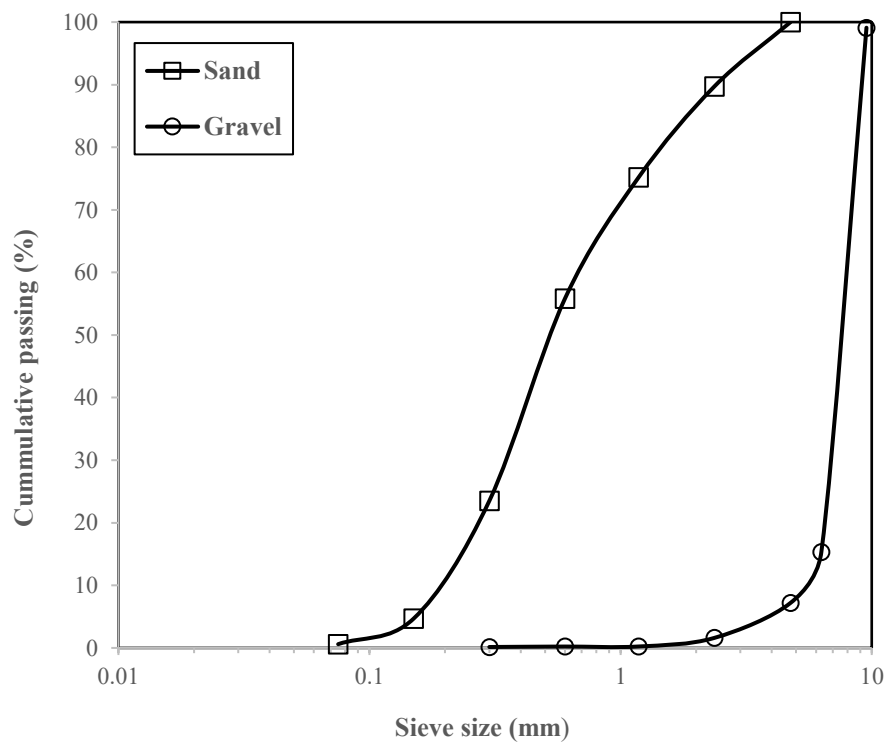
The ordinary Portland cement used was CEM I 52.5R with the mineral composition of 62.4% C_3S , 10.3% C_2S , 10.6% C_3A , 2.2% C_4AF and 4.9% calcite as limestone filler. The oxide compositions of CEM I are presented in Table 3-1. Loss on ignition and specific gravity of CEM I was 2.1% and 3.0998 respectively. Aggregates used were Thames Valley sand and gravel with a maximum size of 5mm and 10mm. The particle size distributions are presented in Figure 3-1. Specific gravity, percentage of moisture absorption to saturated surface dry condition (SSD) and total evapourable moisture content of aggregates were measured with procedures provided by ASTM C566 and C128 (2016; 2019) and presented in Table 3-2.

Table 3-1 Oxide composition of CEM I

Oxide composition: %							
CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃
63.4	20.8	5.4	2.4	1.5	0.3	0.7	2.9

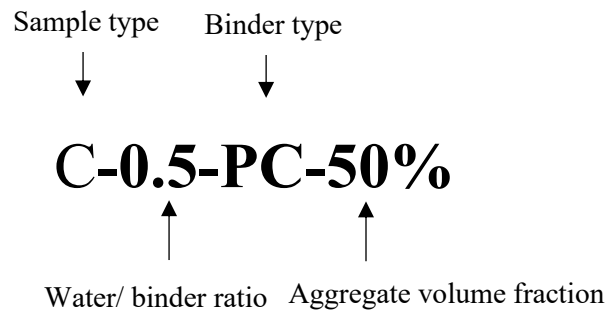
Table 3-2 Aggregate properties

Aggregate type	MSA: mm	Specific gravity	Moisture absorption SSD: %	Moisture content %
Fine	5	2.66	1.05	0.24
Coarse	10	2.51	2.0	0.37

**Figure 3-1 Grading curves for Thames valley sand and gravel.**

3.1.2 Mix proportions

The absolute volume method was adopted to calculate the proportions of CEM I, tap water and aggregates. Generally, cement paste, mortar and concrete samples were cast at a water/binder (w/b) ratio ranging from 0.35 – 0.6 to create samples with low and high porosity. Aggregate volume fractions were adjusted at 20 % to 60 % for mortar and 70 % to 73 % for concrete. To compensate for the difference in moisture content and storage conditions of aggregates, the percentage of moisture absorption to saturated surface dry condition (SSD) and total evapourable moisture content of aggregates were determined based on ASTM C566 and C128 (2016; 2019). Additional moisture was added to the batch water to achieve the designed free w/b ratio. Samples were exposed to moist curing in a fog room at 20 ± 2 °C and 95 ± 5 % RH. Throughout this thesis, samples will be designated using the format presented in Figure 3-2. Specific mix proportions, casting, curing and conditioning procedures were adjusted based on the purpose of the study and will be introduced separately in the respective chapters.



Notation:

	P	=	Paste
Sample type	M	=	Mortar
	C	=	Concrete
Binder type	PC	=	CEM I

Figure 3-2 Sample designation.

3.1.3 Sample conditioning

Vapour diffusivity, permeability and water sorptivity are affected by the moisture content of samples. Thus, samples must be conditioned to a standard moisture state before water absorption and desorption testing. Findings presented in Chapter 6 had shown that changes in relative humidity and water sorptivity are dependent on conditioning humidity and temperature. Hence, conditioning temperature and relative humidity were carefully considered before conducting experimental studies. Furthermore, it would typically require at least 6 months for samples to reach moisture equilibrium by gently drying at 50 % RH and 20 °C (Hong Seong Wong 2006). To lessen conditioning times, samples were conditioned at 55 % RH, 65 % RH, 75 % RH, 86% RH, 93 % RH and 50 °C.

Before conditioning samples, fresh saturated salt solutions were placed in incubators with a motorized fan to generate air circulation. Internal relative humidity was monitored with a Fisherbrand Traceable thermometer and humidity monitor and allowed to stabilize for seven days. Subsequently, samples were weighed and placed in incubators. The percentage mass loss of each sample was carefully measured daily till a mass change smaller than 0.01% was obtained.

3.2 Scanning electron microscope (SEM)

A RFID MEMS sensor, STMicroelectronics HTS221, was chosen based on the specified sensing and design requirements to monitor relative humidity within a range of 70 – 90 % for steel reinforcement with a minimum diameter of 8 mm and provide the ability to quantify and characterize macroscopic interfacial voids (4.2). The sensor consists of a polymer dielectric planar capacitor structure with a silicon exposed material that is prone to degradation after the exposure to high alkaline environments. Furthermore, specimens dried to equilibrium at either 50 °C, 55% and 75% RH can produce varying degrees of microcracks. Surface and morphological changes of described issues can be examined with scanning electron microscope (SEM) to evaluate the suitability of RFID MEMS sensors and explain water absorption and desorption attributes. Additionally, procedures for sample preparation were carefully to prevent any damage before SEM-BSE and EDS characterization (3.2.2).

3.2.1 Principle

Scanning electron microscope (SEM) is an advanced analytical tool that utilizes electrons to observe and evaluate organic and inorganic materials at the micrometre and nanometre scale under vacuum (Egerton, Li, and Malac 2004). Finely focused electron beams will be projected on the surfaces of the sample and emitted back with various energies and signals. This provides information on the surface morphology, crystallographic structure and composition (Goldstein et al. 2007). Notably, low-energy secondary electrons and backscattered electrons are the primary imaging signals used to study topographical changes with an Everhart-Thornley detector (ETD) and through the lens (TLD) (Reimer 2013; T E Everhart and R F M Thornley 1960). Other individual components consist of an electron gun, condenser lens, objective lens, sample chamber, detectors, amplifier and a viewing system shown in Figure 3-3. Furthermore, the spatial distribution of chemical elements can be quantified with energy dispersive spectroscopy (EDS) by gathering characteristic X-rays.

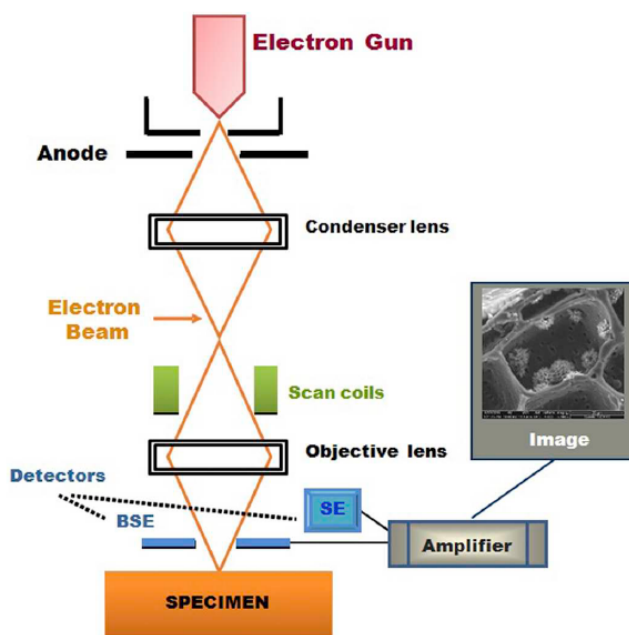


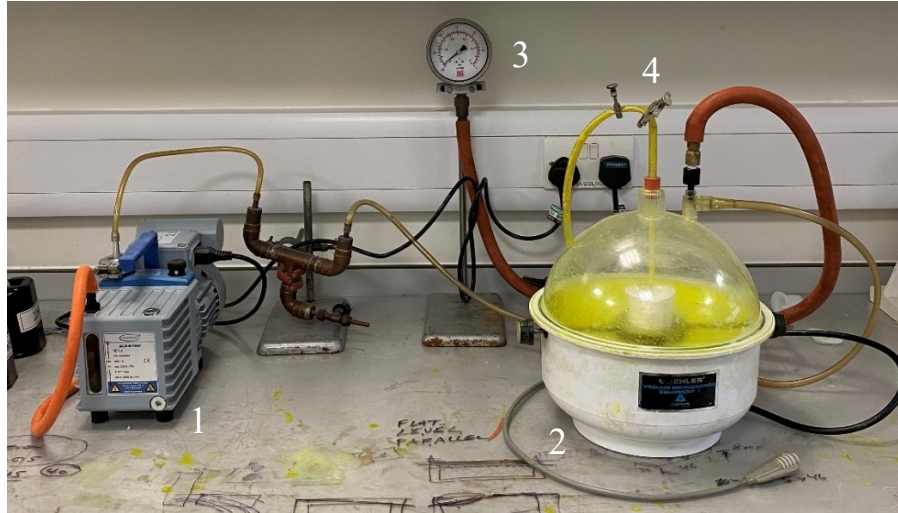
Figure 3-3 Schematic drawing of scanning electron microscope (Kwiecińska, Pusz, and Valentine 2019)

3.2.2 Sample preparation

3.2.2.1 Cementitious material

Cementitious materials were prepared with the following methods developed by Wong & Buenfeld (H S Wong and Buenfeld 2006).

1. **Encasing.** After exposure to cyclic wetting and drying, cement paste samples with the dimensions of $50 \times 50 \times 25$ mm were encased in a silicon rubber mould with the dimensions of $65 \times 65 \times 30$ mm to prevent damage in the following steps. The surface of interest was preserved with the application of water-proof tape and double-sided tape. Subsequently, epoxy resin manufactured by West System was poured into moulds to a depth of 10 mm and allowed to harden for 24 hours (Figure 3-6).
2. **Impregnation.** After demoulding from silicon rubber moulds, tapes and any residual epoxy were removed from the surface of interest. Specimens were transferred into a pressure chamber and placed under vacuum for 1 hour to remove air in pores shown in Figure 3-4. Subsequently, Struers EpoFix resin with hardener at a mass ratio of 22:3 was mixed with 3 % wt. toluene and poured onto the sample surface. The chamber was depressurized to allow the resin to flow into pores and cracks. This is followed by transferring samples to a pressure chamber at 3 bars for deeper impregnation (Figure 3-5).
3. **Cutting.** After 24 hours, specimens were retrieved from the pressure chamber and blocks with the size of $10 \times 50 \times 25$ mm were extracted using a Buehler Isomet 1000 precision cutter at a speed of 180 rpm to prevent any damage shown in Figure 3-7. Moisture was removed from samples by drying in an oven at 50°C till a mass change of less than 0.01% for 5 days.
4. **Grinding.** The surface of interest was ground using SiC papers of successively finer grit sizes of 220 and 1200 to remove residual epoxy and rough surfaces. After grinding, samples were cleaned with acetone in an ultrasonic bath and stored in a desiccator at 3% RH and 21°C containing silica gel for 2 days to remove excess moisture before SEM-BSE characterisation.



Notation:

- | | |
|-------------------|-------------------|
| 1. Vacuum pump | 3. Pressure gauge |
| 2. Vacuum chamber | 4. Clamps |

Figure 3-4 Equipment for vacuum epoxy impregnation.



Notation:

- | | |
|-------------------|-------------------|
| 1. Vacuum chamber | 3. Gas cylinders |
| 2. On-off valve | 4. Pressure gauge |

Figure 3-5 Equipment for deep impregnation.

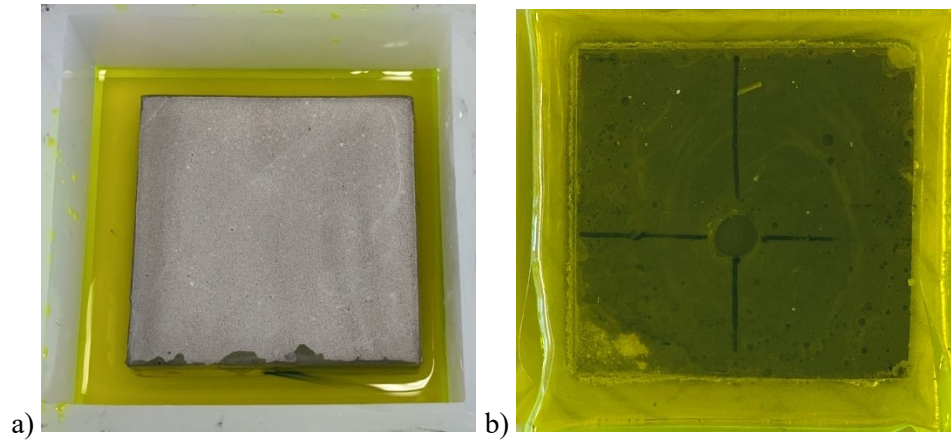


Figure 3-6 Cement paste specimen with a 6.5 mm diameter air void placed a) in $65 \times 65 \times 30$ mm mould for encasing and b) vacuum impregnated in a pressure chamber at 3 bars.

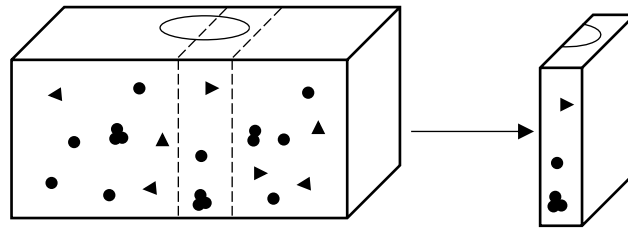


Figure 3-7 Extracting $10 \times 50 \times 25$ mm block samples marked with dotted lines to examine void-paste interface with SEM-BSE.

3.2.2.2 RFID MEMS sensor

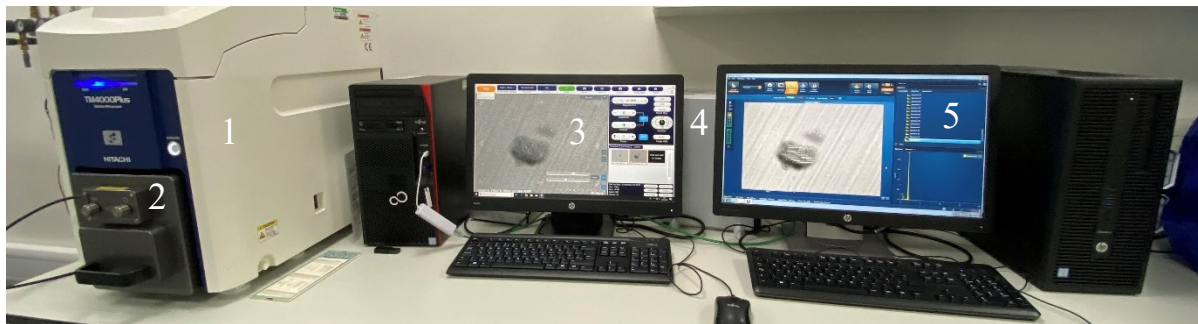
RFID MEMS sensors were prepared with the following methods.

1. **Retrieval.** After exposure to 3% wt. NaCl salt solution, 100% RH and embedment in concrete for 7 months dust particulates on the surface of RFID MEMS sensors were removed with compress gas (RS PRO air duster) and examined with an optical microscope (Olympus BX 51).

2. **Drying.** RFID MEMS sensors were transferred to a desiccator at 3% RH and 21°C containing silica gel for 2 days to remove excess moisture before SEM-BSE and SEM-EDS characterization.

3.2.3 Instrument

Morphological changes and elemental analysis of RFID MEMS sensor, electronic components and paste-void interface were examined with a Hitachi TM4000 Plus scanning electron microscope (SEM) equipped with an Oxford Instruments Aztec Advanced X-Act Compact energy dispersive X-ray detector (EDS) shown in Figure 3-8. The SEM was operated at 15 kV in backscattered electron (BSE) mode with a working distance of 7 mm to achieve an optimal spatial resolution (Yio, Wong, and Buenfeld 2016).



Notation:

- | | |
|-------------------------------|-------------------------------|
| 1. Main unit | 3. Viewing screen for SEM-BSE |
| 2. Sample chamber | 4. X-ray detector |
| 5. Viewing screen for SEM-EDS | |

Figure 3-8 Hitachi TM4000 Plus scanning electron microscope.

3.3 Stereomicroscope

Morphological changes of silicon thin films and air voids were examine and captured with stereomicroscope to provide a quantitative comparison along side SEM-EDS characterization. It is non-destructive and effective technique that does not require the removal of excess moisture before examination. Captured images can be further processed in Fiji (ImageJ version 1.53t NIH) with known luminance value to obtain meaningful quantitative data (3.3.4).

3.3.1 Principle

Stereomicroscopes are designed to provide three-dimensional images of objects with similar mechanisms and stereoscopic vision to the human eye (Miyake et al. 2019). This is achieved by projecting stereoscopic images of objects with independent optical systems. Currently, the two primary stereoscopic systems are the common main objective (CMO) and Greenough shown in Figure 3-9. The Greenough stereomicroscope consists of two separate tilted zoom systems that are suitable for capturing images with a smaller working distance. In comparison, the common main objective stereomicroscope (CMO) has the zoom optical system positioned in parallel to the objective lens which is capable of operating with a longer working distance. However, images are typically distorted due to non-paraxial optics (Hu et al. 2017).

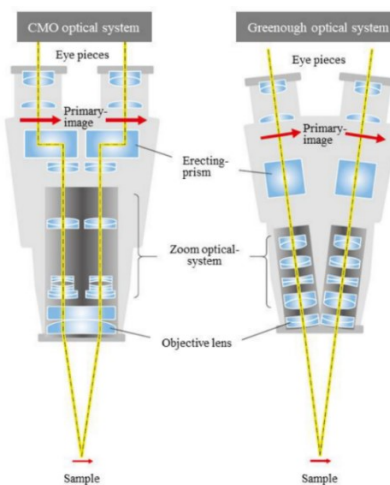
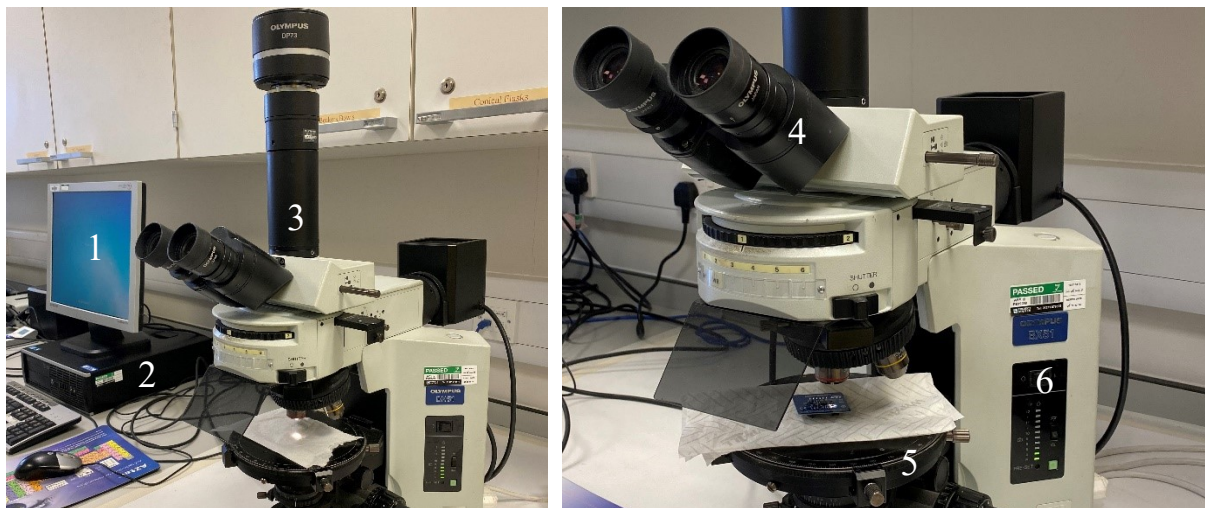


Figure 3-9 Schematics of common main objective (CMO) and Greenough optical systems (Miyake et al. 2019)

3.3.2 Olympus BX51 stereomicroscopy

Morphological changes of silicon thin film were examined with an Olympus BX51 stereomicroscope equipped with a DP73 high colour sensitivity camera capable of capturing images at low light shown in Figure 3-10. Prior to examining specimens, excess dust and debris were removed with the application of compressed gas with an RS PRO air duster. The light intensity was adjusted to a voltage of 6 V and observed at 5x magnification for optimal imaging conditions. To characterize and quantify the rate of degradation, images were captured and stored in a portable storage device. Subsequently, images were imported and further processed with Fiji (ImageJ version 1.53t NIH) (Schindelin et al. 2012; Tan et al. 2016).



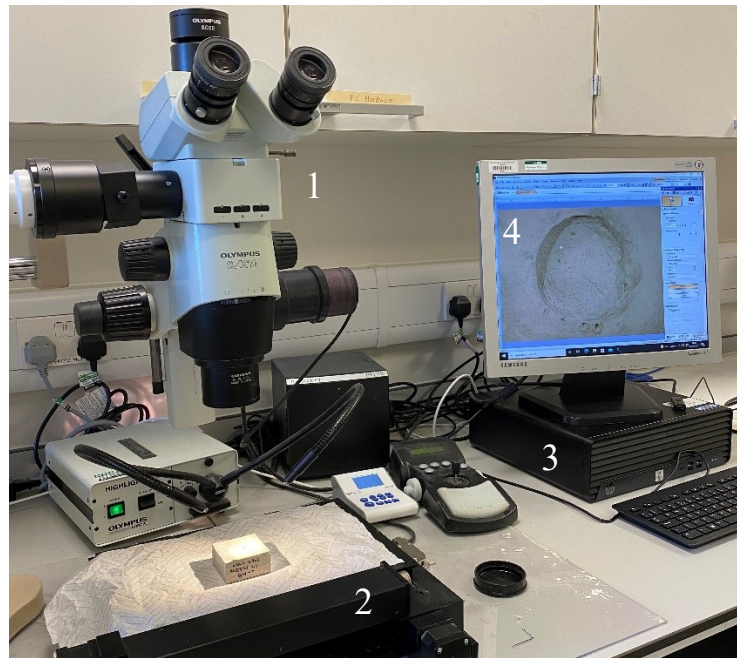
Notation:

- | | |
|---------------------|-------------------------------------|
| 1. Viewing screen | 3. Stereomicroscope |
| 2. Computing device | 4. Lenses |
| 5. Mechanical stage | 6. Microscope control circuit board |

Figure 3-10 Olympus BX51 stereomicroscope.

3.3.3 Olympus SZX10 stereomicroscopy

Morphological changes of air voids on cement paste samples were examined with an Olympus SZX10 stereomicroscope equipped with an Olympus SC50 camera shown in Figure 3-11. To investigate and characterize changes in surface morphology, images were captured prior to conditioning samples in incubators and oven. Subsequently, images of air voids were recaptured after subjecting samples to wetting and drying cycles for comparison. During examination, magnification was adjusted between 0.63x to 6.3x for optimal imaging conditions. Captured images were stored in a portable storage device and imported into Fiji (ImageJ version 1.53t NIH) (Schindelin et al. 2012) for further analysis.



Notation:

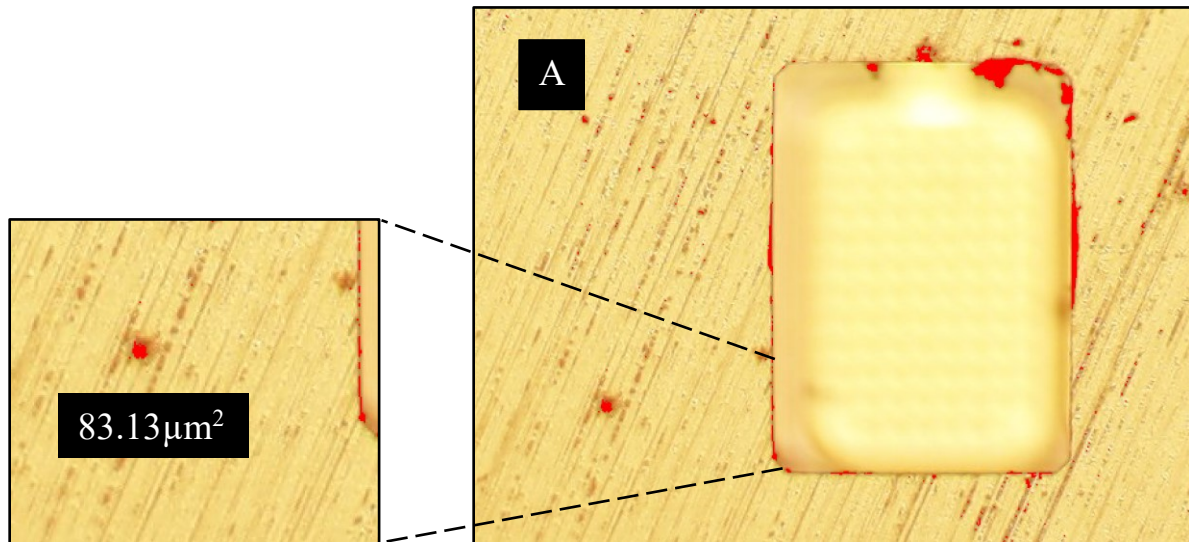
- | | |
|---------------------|---------------------|
| 1. Stereomicroscope | 3. Computing device |
| 2. Moving stage | 4. Viewing screen |

Figure 3-11 Olympus SZX10 stereomicroscope.

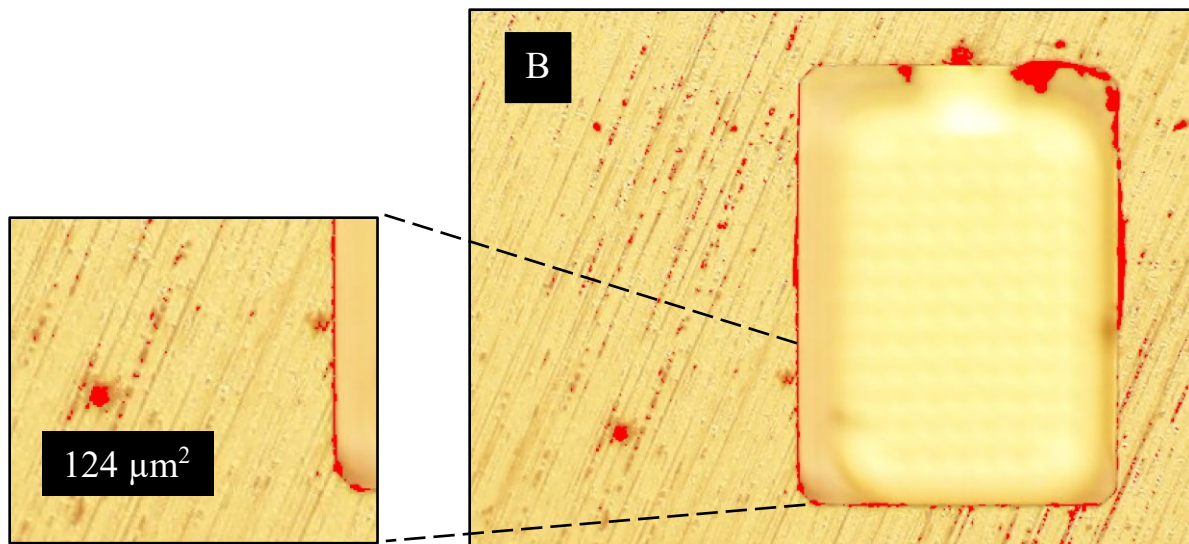
3.3.4 Adjusting colour threshold for degradation analysis

The process of utilising available information within an image for degradation analysis begins by creating a pixel-intensity histogram from a standard image. For a 8-bit image, this requires analysing the number of pixels at each of the 256-pixel intensities. This could be done by calling the 'histogram' function in Fiji (ImageJ version 1.53t NIH) that creates a cumulative threshold spectra (CTS) by determining the percentage of pixels in each pixel intensity (Johnson and Walker 2015). A luminance value of 0 represents pixels that are pitch-black, 255 represents bright pixels and the remaining values (1 to 254) represent pixels with increasing brightness.

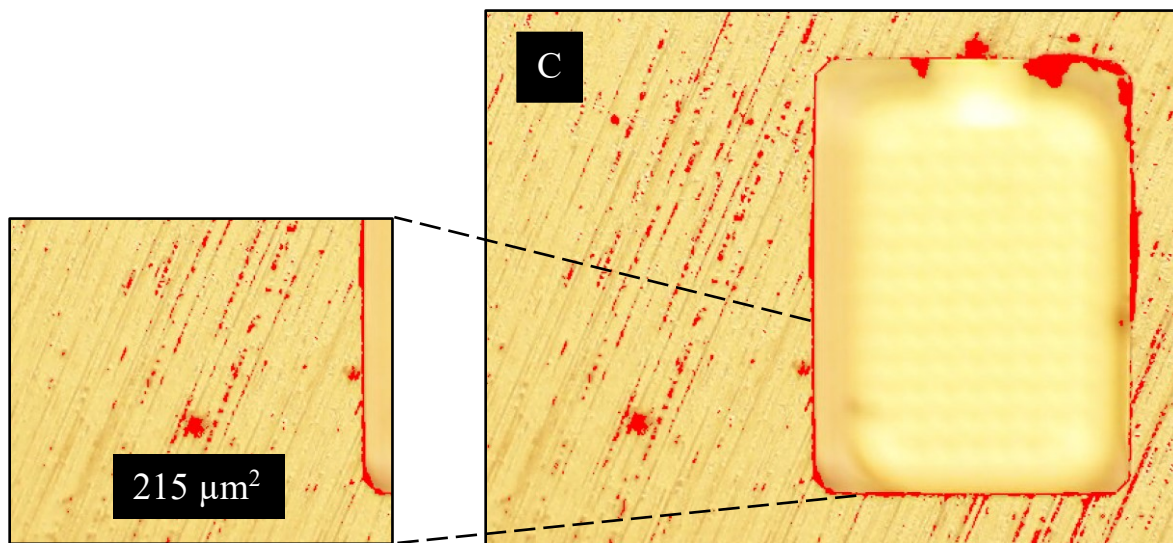
Although images were captured with similar brightness and contrast, the variation in luminance value can overestimate or underestimate degradation areas. More importantly, for quantitative analysis, a luminance value must be predetermined for every image captured, so that meaningful quantitative data could be obtained. Figure 3-12 presents the estimated area of degradation at insufficient illumination (A) with a luminance value of 180, sufficient illumination (B) with a luminance value of 195 and over-illumination (C) with a luminance value of 210. It was shown that examining degradation areas with improper illumination could result in a maximum difference of 74%.



a) Insufficient illumination



b) Sufficient illumination



c) Over illumination

Figure 3-12 The influence of luminance values on estimated degradation area.

3.4 Chapter Summary

In this chapter, materials, experimental procedures and instrumentation used in this thesis were described. Mix design, curing and conditioning regimes were adjusted based on the purpose of the study and will be presented in the respective chapters. Methods used to preserve and examine cracks at the paste-void interface are adopted from well-established procedures. However, additional care was taken when extracting block samples to prevent any additional damage.

SEM-BSE and SEM-EDS are advanced analytical techniques capable of evaluating samples at the micrometer scale. However, with incorrect adjustments to luminance values, the rate and area of degradation of silicon thin films and electronic components could be over or underestimated. Discussions on the influence of luminance values on estimated degradation area are given. Notably, the reliability of 3D printed enclosures plays a crucial role in providing a suitable technique for monitoring relative humidity at the steel interface. Further design considerations and optimisation of enclosures will be described in the next chapter.

Chapter 4 Developing a novel non-destructive method to monitor relative humidity at the steel-concrete interface using RFID-MEMS sensors

This chapter reports on the suitability and feasibility of embedding wireless sensor technologies in customized enclosures to monitor changes in relative humidity at the steel-concrete interface. Considerations of design requirements, commercial availability, technical specifications, principle and application are also discussed. Methods to determine the influence of moisture and aggregate content on transmitted power were developed by conditioning mortar and cement paste samples at 50 °C and 75% relative humidity then submerging them in tap water until a constant weight was achieved. It was shown for the first time that RFID MEMS sensors are capable of monitoring relative humidity changes at the steel-concrete interface for samples with different curing ages, nominal cover depths and water to cement ratio, demonstrating the suitability of such a device for laboratory-based research. Relative humidity measurements, communication range and transmitted power were examined to better understand the influence of aggregate content and degree of saturation. The impact of enclosure designs on particulate contamination and degradation of RFID MEMS sensors are discussed.

4.1 Introduction

At the steel-concrete interface, the presence of moisture can considerably influence the durability performance of reinforced concrete (2.2). Therefore, the ability to characterize and monitor moisture content at the steel-concrete interface is highly valuable for many research and field applications. However, a review of the literature has indicated a lack of non-destructive methods for direct monitoring and evaluation.

This study aims to develop a suitable method for monitoring relative humidity at the steel-concrete interface with RFID MEMS sensors. To comply with British codes and standards, loading tests were conducted on the designed enclosure in accordance with BS 7973-1 (Standard 2001). The influence of

cementitious materials and moisture content on wireless communication range and transmitted power was studied by soldering wires onto the sensor board and monitored with a Solartron 7050 digital multimeter. Relative humidity changes at the steel interface were monitored with samples of different nominal cover depths and curing ages.

4.2 Sensor selection

Before conducting a comprehensive search of commercially available relative humidity sensors and electronic circuit boards, design considerations and sensing requirements were specified and listed in Table 4-1. The dimensions of electronic circuit boards have to be smaller than $50 \times 50 \times 50$ mm to embed sensors in enclosures with a minimum cover depth of 50mm. The electrically erasable programmable read-only memory (EEPROM) will require to have a minimum of 1 Kbyte of data to allow users to monitor measurements on a timely basis. This is based on the assumption that relative humidity and moisture content fluctuates at a significant slower rate in real structures and can be monitored at hourly intervals. For example, surfaces 80 mm above the mean sea level with a 70 mm influential depth of moisture transport is predicted have an absorbed moisture content of $2.6 \times 10^{-3} \text{m}^3$ and a maximum internal relative humidity of 80 % was observed for 50 mm thick concrete samples exposed to water for a period of 4 weeks (J., W., and M. 2001; Padala, Bishnoi, and Bhattacharjee 2022). Additionally, measurements have to be transmitted by wireless communication to ensure that structural integrity and durability are unaffected.

With the aforementioned requirements and considerations, it is also critical to consider the design and sensing specifications of relative humidity sensors. This plays a crucial role in determining the feasibility of monitoring relative humidity at the steel-concrete interface and interfacial voids. Dimensions of relative humidity sensors have to be smaller than $5 \times 5 \times 5$ mm to accommodate a wide range of steel reinforcement including steel reinforcement with a minimum diameter of 8 mm. Particularly, quantification within the millimeter range is required to characterize moisture content in macroscopic interfacial voids. Furthermore, for laboratory-based research, a response time of less than 60 seconds will be desirable to detect instantaneous changes in relative humidity. A sensing range between 70 – 90 % is required to monitor the depassivation of steel during carbon-induced and chloride-induced corrosion (2.2). Lastly, the internal temperature of reinforced concrete structures can range between -5 – 40 °C

during the summer and winter seasons (Brownjohn and Moyo 2001; Prakash et al. 2018). Hence, sensors must operate at a minimum temperature range of -10 – 50 °C.

Table 4-1 Design considerations.

Criteria	Essential	Desirable
Electronic circuit board		
Size and dimensions	< 50mm	1 Kbyte data Electrically Erasable Programmable Read-Only Memory (EEPROM)
Data storage		
Data transmission		
	Wireless communication, 13.56 MHz high frequency (HF) range	
Sensor chip		
Size and dimension	< 5×5 ×5 mm	< 60 seconds
Accuracy		
Response time		
Operating conditions		
Sensing range		
	± 5% RH	
	-10 – 50 °C	
	70 – 90 % RH	

4.2.1 Microchip Technology HS1101LF capacitive humidity sensor

4.2.1.1 Sensor design

The HS1101LF capacitive humidity sensor manufactured by Humirel has the capability of reducing moisture condensation after long periods in saturation phase with an accuracy of $\pm 2\%$ RH, deviation of $\pm 0.5\%$ RH per year and humidity hysteresis of $\pm 1\%$ RH specified by the manufacturer. The relatively low humidity hysteresis value indicates a good repeatability of the sensor, which is determined by measuring the maximum difference in measurements when exposed to wet and dry environments. Shown in Figure 4-1, the overall dimensions consist of a 10mm diameter and 6mm depth encasing with 12.2mm long wires

for attachment on electronic circuit board. The sensor is constructed with a water absorbent dielectric material placed between two electrodes and relative humidity is determined by changes in relative permittivity of the material (Figure 4-1 b). Additionally, it operates at a temperature range of -60 – 140 °C.

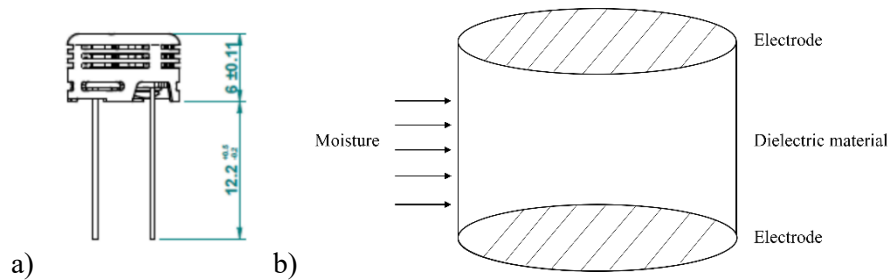


Figure 4-1 HS1101LF relative humidity sensor manufactured by Humirel with a) schematics and b) sensing principle.

4.2.1.2 Hardware and electronic components

To monitor relative humidity, the HS1101LF capacitive humidity sensor is attached to a PICTail™ Demo board. Changes in capacitance are measured by linking an operational amplifier (op amp) and resistor which acts as an inverting (Miller) integrator which produces an output voltage that is proportional to the integral of the input voltage. During measurements, a square wave is transmitted from the microcontroller to the integrator and converted to a triangle wave output. The time required for the triangular wave to rise and fall is determined by the microcontroller and converted to a capacitance value. Measurements can be transferred to a personal computer with the aid of a PICKIT™ Flash starter kit and USB cable shown in Figure 4-2.

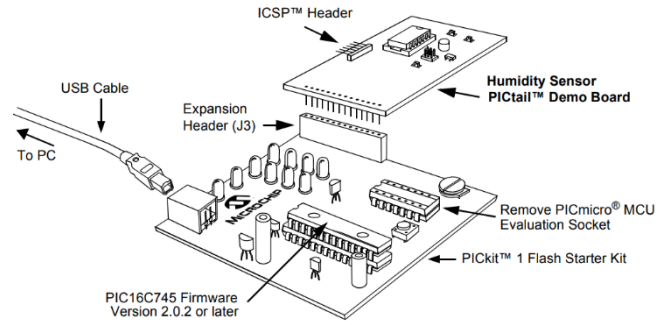


Figure 4-2 Schematics of Humidity Sensor PICtail™ Demo board with the dimensions of 65×50 mm attached to a PICkit™ Flash starter kit to evaluate relative humidity measurements.

4.2.2 Renesas HS3000 humidity sensor

4.2.2.1 Sensor design

The HS3000 microelectromechanical systems (MEMS) humidity sensor manufactured by Renesas has an integrated calibration and temperature compensation logic that corrects relative humidity and temperature measurements without the requirement for users to calibrate output data. It is capable of operating at a humidity and temperature range of 0 to 100 % and $-40 - 125$ °C with an accuracy of ± 1.5 % RH, deviation of ± 0.25 % RH per year and humidity hysteresis of ± 1 % RH specified by the manufacturer. Shown in Figure 4-3, the sensor is constructed with a $3 \times 2.41 \times 0.8$ mm encasing with cantilever beams coated with an expanding nano-water vapour polymer film. An increase in moisture content induces stress and strains on the cantilever beam that can be monitored with embedded resistors.

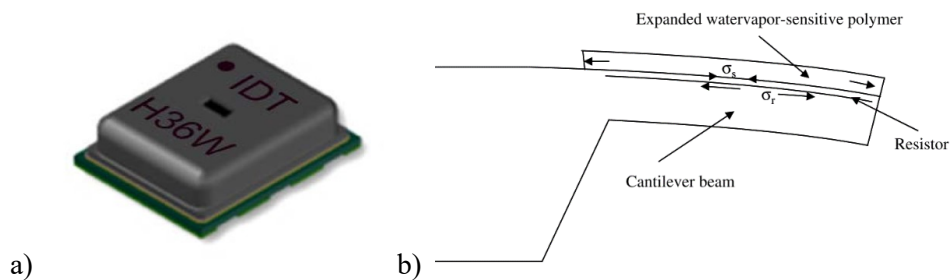


Figure 4-3 HS 3000 humidity sensor manufactured by Renesas with a) schematics and b) sensing principle.

4.2.2.2 Hardware and electronic components

The SDAH01 Evaluation Kit enables users to evaluate the performance of the HS3000 relative humidity and temperature sensor, and perform data logging experiments through a standard USB interface with a demo software. From the software interface, users can observe measurements in real-time, adjust sensor resolution and configure measured parameters. In addition, the evaluation kit can be powered with a CR 1632 coin cell battery for applications which require continuous monitoring.

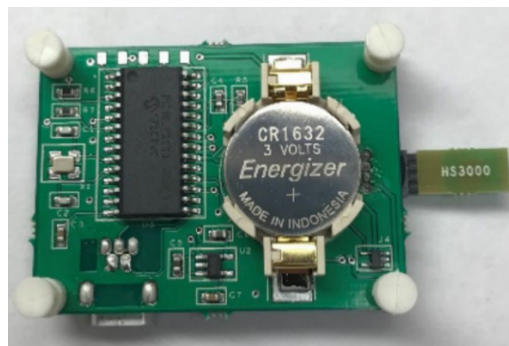


Figure 4-4 Schematics of Renesas SDAH01 Evaluation Kit with the overall dimensions of 44×32 mm and HS 3000 humidity sensor attached.

4.2.3 Texas Instruments HDC2010 humidity and temperature sensor

4.2.3.1 Sensor design

The HDC2010 humidity and temperature sensor is constructed with an ultra-compact Wafer Level Chip Scale Package (WLCSP) that packages an integrated circuit at the wafer level without the requirement for assembling individual components. Furthermore, the sensing element is placed at the bottom to prevent direct exposure to dirt, dust and other environmental contaminants, and has an integrated heating element to dissipate moisture for increased reliability. During manufacturing, the sensor is factory calibrated to have an accuracy of ± 2.0 % RH, deviation of ± 0.25 % RH per year and humidity hysteresis ± 1 % RH specified by the manufacturer. The overall dimensions consist of a $1.5 \times 1.5 \times 0.675$ mm encasing, and

operates at a temperature range of -60 – 140 °C. The sensor is constructed from a similar structure presented in Section 4.2.1.1 with a polymer dielectric material embedded between two electrodes.



Figure 4-5 Schematic of HDC2010 low-power digital relative humidity sensor manufactured by Texas instruments.

4.2.3.2 Hardware and electronic components

The Texas Instruments TIDA-01477 Humidity and Temperature Sensor Node consist of an ultra-low power wireless microcontroller (MCU) platform, a nano-power system timer and a humidity and temperature sensor. This allows measurements to be monitored and transmitted on a timely basis to a sensor-cloud gateway to be stored on a remote storage system. To extend battery life, the nano-power system timer is capable of waking up the system at longer intervals. Depending on the use case, the sensor node can be converted into two configurations. For a sensor-to-cloud configuration, the sensor node will be in time sync with a gateway to transmit humidity and temperature measurements. For long-term monitoring, the sensor node can be integrated with an ultra-low power sub 1 GHz star network, and measurements can be obtained over a 10 year period with a CR2032 lithium-ion coin cell battery.



Figure 4-6 Schematics of Texas TIDA-01477 Humidity and Temperature Sensor Node with the overall dimensions of 76.2 × 38.1 mm and HDC2010 humidity sensor attached.

4.2.4 Sensirion SHT4x relative humidity and temperature sensor

4.2.4.1 Sensor design

The SHT4x relative humidity and temperature sensor is fabricated in an open-cavity dual flat no lead (DFN) package with overall dimensions of $1.5 \times 1.5 \times 0.54$ mm (Figure 4-7). It is based on a polymer capacitive humidity sensor that absorbs or releases moisture in proportion to relative humidity which influences capacitance (4.2.1.1). The sensing element consists of silicon hosted on a copper lead frame encased with an epoxy-based compound. The exposed underside of the lead frame is coated with Ni/Pd/Au. The integrated heating element can be activated with a set of commands to dissipate condensation for increased reliability. During manufacturing, the sensor is factory calibrated to have an accuracy of $\pm 1.0\%$ RH, deviation of $\pm 0.2\%$ RH per year and humidity hysteresis $\pm 0.8\%$ RH specified by the manufacturer. Additionally, it operates at a temperature range of $-40 - 125$ °C.

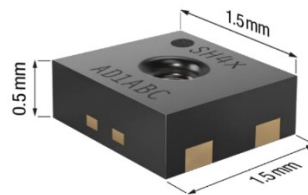


Figure 4-7 Schematics of SHT4x relative humidity and temperature sensor manufactured by Sensirion.

4.2.4.2 Hardware and electronic components

The Sensirion Smart Gadget SHT4x is a reference design circuit board that is equipped with a Liquid Crystal Display (LCD), SHT4x relative humidity and temperature sensor, Bluetooth 4.0 module, microcontroller and battery holder. Users will be able to receive humidity measurements in real time over the 2.402-2.481 GHz wireless frequency range via a mobile application.



Figure 4-8 Schematics of Sensirion Smart Gadget with the overall dimensions of 75×32 mm and SHT4x humidity sensor attached.

4.2.5 STMicroelectronics HTS221 capacitive digital relative humidity and temperature sensor

4.2.5.1 Sensor design

The HTS221 capacitive digital relative humidity and temperature sensor is assembled in a small top-holed cap land grid array (HLGA) package based on a die capped formed with a film-assisted mould alongside an exposed pressure port. The sensing element consists of a conductive polymer with dielectric materials surrounded by a metallic and insulating layer (Figure 4-9 b). In the case of condensation, the integrated heating element can be operated by an external controller to dissipate moisture. The sensor is factory calibrated to have an accuracy of $\pm 3.5\%$ RH, deviation of $\pm 0.5\%$ RH per year, humidity hysteresis $\pm 1\%$ RH and sensitivity of 0.004% RH/LSB (least significant bit) specified by the manufacturer. The overall dimensions consist of a $2 \times 2 \times 0.9$ mm encasing and operate at a temperature range of $-40 - 120$ °C (Figure 4-9 a).

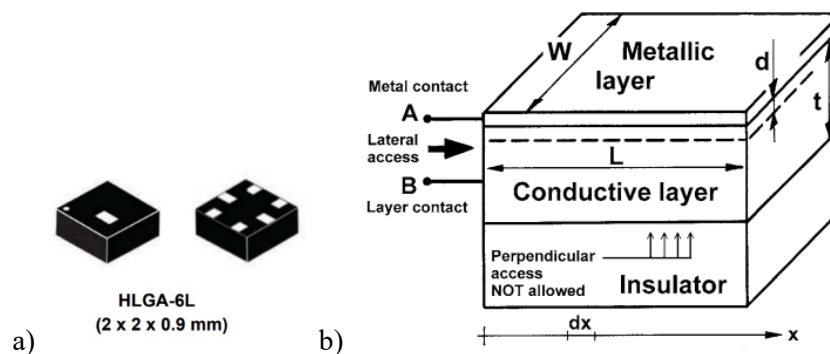


Figure 4-9 HTS221 capacitive digital relative humidity and temperature sensor with a) schematics and b) sensing principle (J I Izpura 2001).

4.2.5.2 Hardware and electronic components

The ST Microelectronics, STEVAL- SMARTTAG1, is a near field communication (NFC) tracker evaluation board that communicates over a set of protocols that transmits and receives information over the 13.56 MHz wireless frequency range between two wireless devices. It utilizes NFC Forum type V based on the RFID technology designated by the ISO/IEC 15693 specification that enables an operational range of 1.5 meters (ISO/IEC 15693-2:2019).



Figure 4-10 Schematic of STMicroelectronics STEVAL-SMARTAG1 with the overall dimensions of 43 × 43 mm.

4.2.6 Summary

Table 4-2 provides an overview of sensor technologies reviewed. A significant proportion of sensors relied on polymer-based capacitor to monitor relative humidity. This sensing principle provides the advantages of a contactless and maskless patterning, low temperature processing and low manufacturing cost (Singh et al. 2010). Nevertheless, polymers have a low relative permittivity in the range of 2 to 4 which limits the sensitivity to moisture changes (Barber et al. 2009). MEMS devices utilizing cantilever beams enables a considerable reduction in size and electric power for actuation, but foreign particles can easily contaminate micro-machines and cause fracture cracks (D. M. Tanner et al. 2000). On the other hand, dielectric planar capacitors integrated with insulators such as ceramic fillers, barium titanate and barium strontium titanate have enhance relative permittivity, sensitivity to moisture changes and mechanical performance (Mikolajek et al. 2019). These attributes can potentially prolong the embedment time in real structures; however, the influence of cementitious materials on performance and durability

must be further investigated. Moreover, by considering the design constraints of electronic circuit boards STEVAL- SMARTTAG1 developed by STMicroelectronics had met the requirements and was selected for further development to monitor relative humidity changes at the steel-concrete interface.

Table 4-2 Overview of sensor technologies and potentials for monitoring relative humidity at the steel interface

Manufacturer	Sensor Dimension (mm)	Hardware Dimension (mm)	Accuracy (% RH)	Transmission	Response time (s)	Sensing principle
Microchip technology, HS1101LF sensor	10 $\varnothing$ $\times$ 18.2	65 $\times$ 50	$\pm$ 2.0	Wired, USB interface	5	Polymer-based capacitor
Renesas, HS3000 sensor	3 $\times$ 2.41 $\times$ 0.8	44 $\times$ 32	$\pm$ 1.5	Wired, USB interface	4	Cantilever structure
Texas Instruments, HDC2010 sensor	1.5 $\times$ 1.5 $\times$ 0.675	76 $\times$ 38	$\pm$ 2.0	Wireless, 1 GHz	8	Polymer-based capacitor
Sensirion, SHT4x sensor	1.5 $\times$ 1.5 $\times$ 0.54	75 $\times$ 32	$\pm$ 1.0	Wireless, 2.402-2.481 GHz	4	Polymer-based capacitor
STMicroelectronics, HTS221 sensor	2 $\times$ 2 $\times$ 0.9	43 $\times$ 43	$\pm$ 3.5	Wireless, 13.56 MHz	10	Dielectric planar capacitor

4.3 Prototype and design

With a comprehensive search from online sources, commercially available enclosures that can accommodate the dimensions of the chosen sensor were found. However, commercially available enclosures are unable to provide direct measurements of relative humidity at the steel surface, as aggregate particles and cement paste can potentially accumulate at the enclosure surface. Nevertheless, it allows an early examination of the functionality of sensors during the prototyping phases. Subsequently, enclosures can be customized and 3D printed to obtain direct measurements at the steel surface.

4.3.1 Commercially purchased enclosures

The feasibility and suitability of monitoring relative humidity were investigated with commercially available polyethylene terephthalate enclosures. The enclosure has the overall dimensions of $45 \times 45 \times 20$ mm with a removable lid to retrieve sensors after embedment. Five one-millimetre diameter holes were drilled at the bottom with an Axminster ED1682 pillar drill with a one-millimetre drill bit attached to allow the ingress of moisture. Subsequently, a waterproof polyester was placed at the bottom of the enclosure to prevent particulate contamination from cement and aggregates.

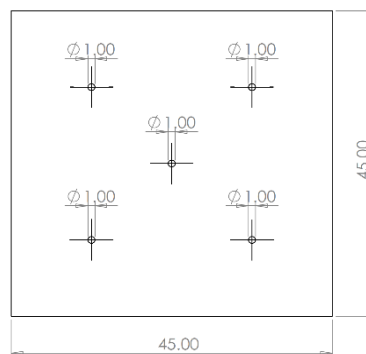


Figure 4-11 Schematics of $45 \times 45 \times 20$ mm polyethylene terephthalate enclosure with five drilled one-millimetre diameter circular holes.

4.3.1.1 Experimentation

Concrete and mortar with a water/cement ratio (w/c) of 0.6 and 0.4, and aggregate volume fractions of 73% and 58% were prepared. Cement I 52.5 R was used with the chemical and physical properties specified in Section 3.1.1. Mixes were designed with the proportions shown in Table 4-3.

Table 4-3 Mix proportions

<i>Mix design</i>	<i>w/c ratio</i>	<i>Water (kg/m³)</i>	<i>Sand (kg/m³) <5mm</i>	<i>Coarse aggregate (kg/m³) <10mm</i>	<i>Cement (kg/m³)</i>	<i>Aggregate vol fraction: %</i>
C-0.6-PC-73	0.6	175.6	750.3	1125.4	292.7	73
M-0.4-PC-58	0.4	235.0	1566.1	-	570.2	58

Four specimens with dimensions of 100 × 100 × 100 mm were prepared. Mortar and concrete were dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Samples were cast in plastic moulds and compacted on a vibrating table until no significant release of air bubbles was observed with polyethylene terephthalate enclosures held in position at 10mm depth from the surface. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of 20±2°C and relative humidity of 95±5% for 2 days. Once the desired curing age has been achieved, specimens were placed in incubators containing saturated ammonium nitrate salts (65% RH) at 72 hours and magnesium chloride salts (33% RH) at 312 and 408 hours.

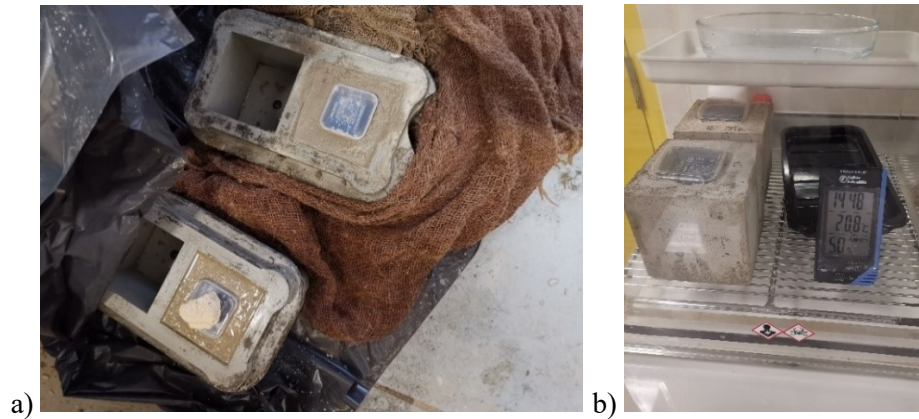


Figure 4-12 RFID MEMS sensors embedded in $100 \times 100 \times 100$ mm concrete and mortar specimens a) wrapped in wet hessian fabric and b) placed in incubators containing ammonium nitrate (65% RH) and magnesium chloride (33% RH) saturated salts.

4.3.1.2 Preliminary result and discussion

From the results presented in Figure 4-13, it was evident that there was a sudden decline in relative humidity at 5.5 hours for mortar specimens during the temperature rise as seen in Figure 4-14. This can be resulted from an increase in water demand for cement hydration after the dormant period (Bullard et al. 2011). It was also speculated that there can be an air void between the enclosure and mortar interface.

Further experiments were conducted by placing specimens in incubators containing ammonium nitrate saturated salts maintained at 65 % RH to observe the effect of drying after 72 hours of curing. Results showed a decrease in relative humidity of 77 % RH and 98 % RH at 86.5 hours for mortar and concrete specimens. These observations are expected because moisture will require a longer time to diffuse from specimens with a higher moisture content. Subsequently, after 150 hours it was observed that internal relative humidity has begun to stabilize and equilibrate. Further gentle drying and wetting tests were conducted firstly by placing samples in incubators containing saturated magnesium chloride salts maintained at 33% RH at 312 hours and 100% RH environment at 360 hours. From the relative humidity measurements obtained, it was observed that specimens with higher water to cement ratio have faster drying rates. These observations are expected because water desorption is dependent on capillary porosity where a higher abundance of empty pores will provide a faster capillary desorption (Wu et al. 2019).

Additionally, sensors were removed from enclosures and samples were sectioned in half with a Buehler Delta automatic abrasive cutter to observe the surface morphology between the enclosure and specimen.

It was found that the mortar specimen had an air void with a length of 28.7mm adjacent to the sensor shown in Figure 4-15. This abnormality could have been caused by improper compaction which had contributed to the decline in relative humidity at 5.5 hours. Nonetheless, this indicates that the RFID MEMS sensor was capable of monitoring relative humidity changes of air voids in cementitious materials. It is important to note that this is applicable under the circumstance that the sensor is directly exposed to an air void. In cases without direct exposure, the sensor might not be able to detect sudden changes in relative humidity.

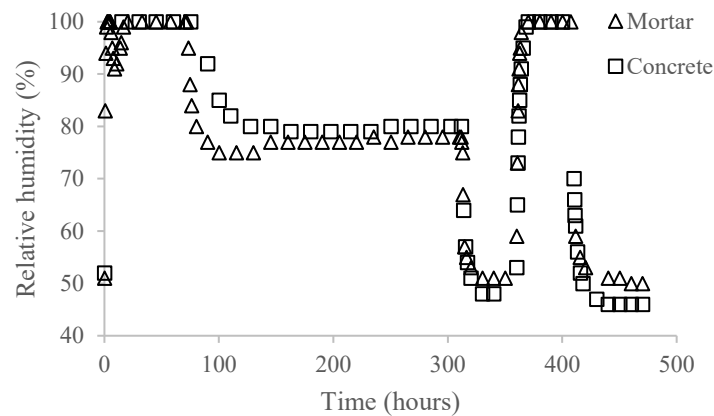


Figure 4-13 Measured changes in the relative humidity of samples in incubators containing saturated ammonium nitrate (65% RH) at 72 hours and magnesium chloride (33% RH) at 312 hours and 408 hours.

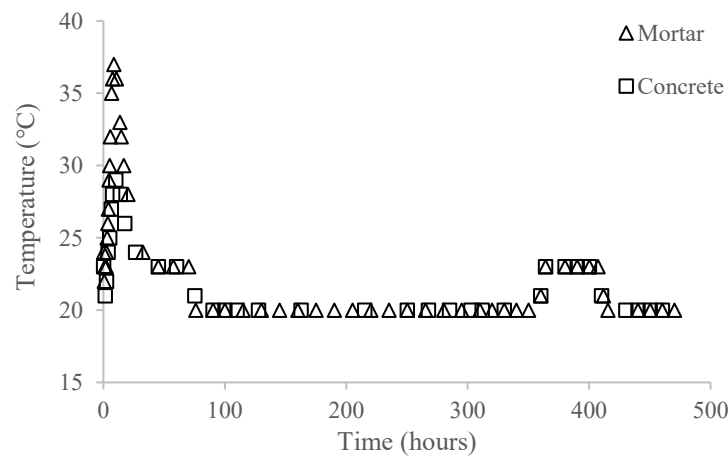


Figure 4-14 Measured changes in temperature of samples in incubators containing saturated ammonium nitrate (65% RH) at 72 hours and magnesium chloride (33% RH) at 312 hours and 408 hours.

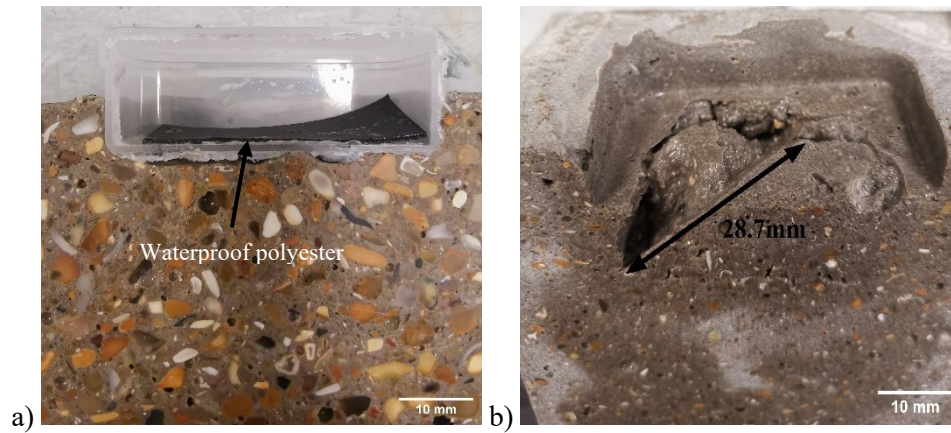


Figure 4-15 Examining the enclosure-specimen interface of a) concrete and b) mortar after wet cutting with Buehler Delta automatic abrasive cutter.

4.3.1.3 Limitations

As mentioned in section 4.3.1.1, RFID MEMS sensor was capable of detecting relative humidity changes of irregularities at the enclosure-specimen interface. However, the design and implementation of commercially available enclosures can affect the reliability and accuracy of measurements obtained. As shown in Figure 4-15 a, waterproof polyester material was placed in enclosures to protect electronic components during concrete casting. Nevertheless, the hysteresis of materials can influence the overall measured accuracy and reliability with a slower diffusion of moisture into the enclosures (Sokoll and Jacob 2014). This also raises the question of whether the measurements obtained are a good representation of the monitored environment. Hence, it would be ideal to embed RFID MEMS sensors in customized enclosures to provide a direct measurement of the steel surface without depending on protective material.

4.3.2 3D printing

4.3.2.1 Software

Customized enclosures for RFID MEMS sensors were sketched and designed with SolidWorks, a CAD software based on parametric design capable of creating 3D models and assemblies. During the prototyping stage, initial sketches were created by measuring the dimensions of RFID MEMS sensors with a vernier calliper and exported as an STL file. This format was required to transform 3D models into G-code that can be readable by 3D printers. The STL file was transferred to a portable device and imported to an Ultimaker S5 pro 3D printer through a USB interface for 3D printing (Figure 4-16).

4.3.2.2 Sample preparation

Prior to 3D printing, factors impacting printing quality were considered. This includes changes in surrounding and chamber temperature that can affect the mesostructure and bond strength of printed parts (Sun et al. 2008). To prevent any fluctuation in temperature, components were printed in a temperature-controlled laboratory at $21 \pm 2^\circ\text{C}$. The material chosen for 3D printing consisted of polylactic acid (PLA) with a diameter of 2.85 mm and a maximum roundness deviation of 0.1 mm. PLA material was considered due to its high commercial availability, and reasonable mechanical properties and tolerances (Mazzanti, Malagutti, and Mollica 2019). Previous studies have shown that PLA Ultimaker filament constructed with Fused Filament Fabrication (FFF) technique has an ultimate tensile strength of 57.15 Mpa and elastic modulus of 2606 Mpa (Morettini et al. 2022).



Notation:

- | | |
|------------------|----------------------|
| 1. Feeder | 3. Printed component |
| 2. Nozzle | 4. Hot plate |
| 5. Control panel | 6. USB interface |
| 7. Filament | |

Figure 4-16 Schematics of Ultimaker S5 pro.

4.3.2.3 Fused deposition modelling technique and design procedures

Fused deposition modelling (FDM) is a common additive manufacturing technique that extrudes and deposits semi-molten thermoplastic filaments in layers through computer numerical control (CNC)

nozzles (Rezayat et al. 2015; Taufik and Jain 2022). During the fabrication process temperature at the nozzle is maintained at a constant temperature which provides sufficient time for the material to be extruded (Livesu et al. 2017). However, machine reliability, process parameters and shrinkage of material during hardening can impact the overall dimensional accuracy and quality of printed parts. Thus, before achieving desired dimension and quality from printed parts, 3D CAD models were calibrated by a simple process and predefined conditions stated below.

3D CAD models were calibrated with the following steps.

1. **Design.** The dimensions of the top and bottom enclosures were drawn to scale on SolidWorks. Subsequently, individual 3D CAD models were exported as an STL file format and stored in a portable storage device.
2. **Print.** The portable storage device was connected to the 3D printer (Ultimaker S5 pro) via a USB interface, and the STL files were imported to a software interface. Prior to printing, appropriate 3D CAD models were selected with specified quantities.
3. **Examination.** Once individual components were printed, the dimensions of 3D printed parts were measured with a vernier calliper and compared against 3D models to inspect for machine reliability. Differences in dimensional accuracy were noted down and 3D CAD models were adjusted to compensate for machine error.
4. **Embedment.** RFID MEMS sensors were placed in printed enclosures and embedded in concrete.
5. **Retrieval.** Once the desired curing age has been achieved, RFID MEMS sensors were recovered from printed enclosures. Particulate contamination and morphological changes of silicon thin films were examined with an Olympus BX51 stereomicroscope.
6. **Calibration.** Dimensions of 3D CAD models were further adjusted to satisfy the predefined conditions stated below. Subsequently, previous steps were repeated with enclosures printed and embedded in concrete for further examination.

The 3D CAD models and enclosures were designed with predefined conditions.

1. **Contamination.** Seepage of concrete during embedment and casting must be prevented with customized enclosures. In addition, no significant particulate contamination should be observed on the surface of silicon thin films.
2. **Direct measurement.** Besides providing sufficient protection, designs of enclosures shall provide direct measurements of relative humidity at the steel-concrete interface.
3. **Dimensions.** The dimensions of enclosures must be smaller than 50×50 mm to replace existing construction tools with a minimum cover depth of 50mm.
4. **Stresses.** While developing 3D CAD models, enclosures should be designed to prevent and reduce stresses induced on the sensor during concrete casting and curing to improve reliability.

4.3.3 Second Prototype

The enclosure consists of a 8.5 mm diameter groove along the sensor exposure surface shown in Figure 4-17 a. After 3D printing, the capability of protecting RFID MEMS sensors during concrete casting was examined by placing 8 mm diameter steel bar above the enclosure. The bottom enclosure was embedded in a silicone mould to retrieve the sensor without damaging specimens using destructive methods. Specimens were cast in plastic moulds and compacted on a vibrating table until no significant release of air bubbles was observed. After compaction, specimens were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. Upon retrieving the sensor, it was discovered that water has penetrated the enclosure during concrete pouring (Figure 4-18). This can cause electronics to fail from electrostatic shock during an electrostatic discharge between sensor components. Furthermore, foreign particles can contaminate the MEMS sensor and impact the accuracy of measurements.

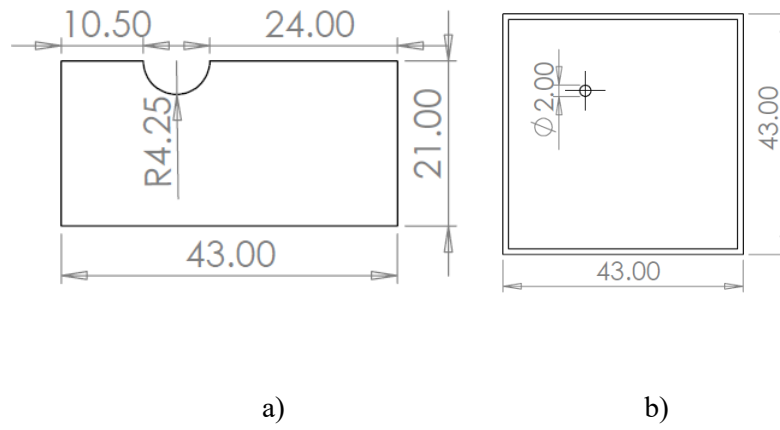


Figure 4-17 Schematics of the second prototype with the a) side and b) bottom view.



Figure 4-18 Water bleeding into the enclosure during concrete pouring

4.3.4 Final design

With the prior issues mentioned in section 4.3.3, a larger diameter groove of 9.5mm was introduced along the sensor exposure surface shown in Figure 4-19 a. Similarly, the design was exported from SolidWorks and 3D printed with Ultimaker S5 pro 3D printer. After casting eight specimens of low and high-strength concrete, it was observed that no water or foreign particulates had penetrated the enclosures. This gave a clear indication that the designed enclosure was able to protect sensors from particulate contamination during concrete pouring. As presented in Figure 4-20, the method can be adopted to 3D print enclosures for the specific purpose of monitoring relative humidity at the steel-concrete interface. It is customizable to suit spacers with a cover depth of 50 mm and 100 mm.

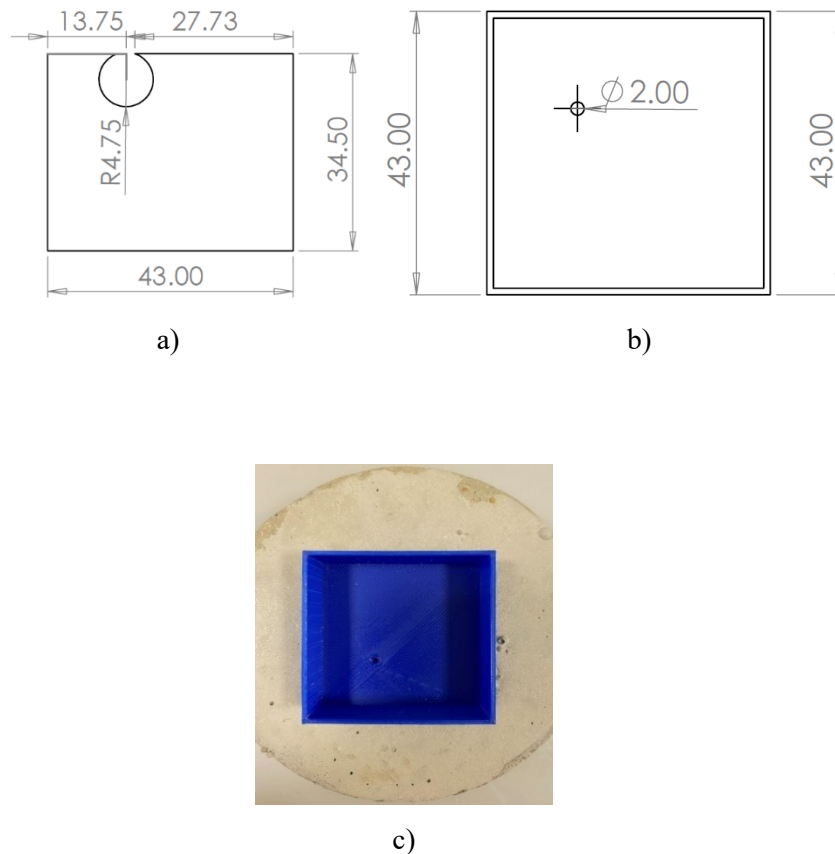


Figure 4-19 Schematics of the final design with the a) side, b) bottom view and c) top view of the sample after 24 hours of curing.

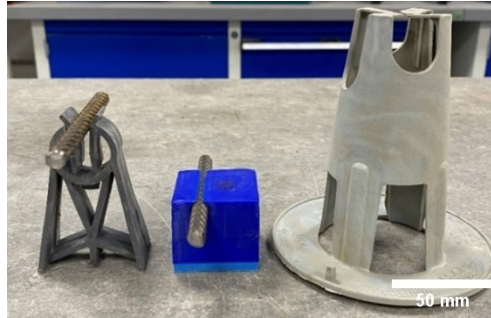


Figure 4-20 Comparing the developed method with commercially available reinforcement spacers for 50mm of cover on the left and 100mm of cover on the right.

4.3.4.1 Loading and deflection tests

To ensure the customized enclosure can withstand loads when cast in concrete, loading tests were conducted in accordance with the British standard BS 7973-1 (2001). As stated in BS 7973-1, the designed enclosure is categorized under the light category for reinforcement with a diameter not greater than 16 mm and cover depth in the range of 15 - 50 mm. Annex A.1.3.2 discloses that the enclosure must be placed on a flat gauge plate during testing. A load of 0.25 kN must be applied within 10 seconds and remain applied for a further 10 mins. After removing the test load, the height of the enclosure must not deflect by more than 1 mm.

To comply with the testing requirements, a 8 mm diameter ribbed steel bar was attached to the 3D printed enclosure with a flat gauge plate placed below the enclosure as shown in Figure 4-21. The initial height was measured with a vernier calliper before conducting the loading test with an Instron testing machine. After applying a load of 0.25 kN for 10 minutes, the enclosure was examined for signs of cracking and the final height was remeasured with a vernier calliper. Tests were conducted on 3 replicates with a maximum deflection of 0.05mm and no signs of cracking.

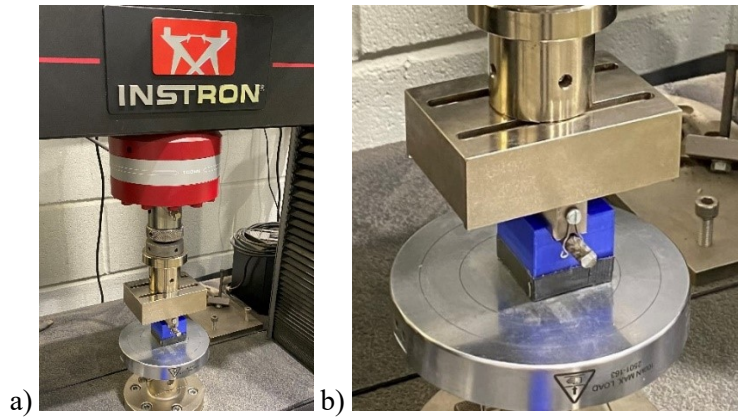


Figure 4-21 Image illustrating test method used to determine the maximum deflection of PLA 3D printed enclosure a) with a 100 KN load cell attached and b) closer image of the test method.

4.4 Testing precision and accuracy of MEMS sensors

As reported by the manufacturer, the overall repeatability of each sensor is $\pm 0.5\%$ RH which appears to be relatively high. Thus, relative humidity from each sensor was monitored in a temperature-controlled laboratory at $21 \pm 2^\circ\text{C}$ and $55 \pm 5\%$ RH. In total, 30 individual sensors were tested and the maximum deviation was $\pm 1\%$ RH. Subsequently, the accuracy of MEMS sensors was tested with incubators and enclosures containing sodium chloride (75% RH), potassium hydrogen sulphate (86% RH), potassium nitrate (93% RH) salt solutions and moisture (100% RH). Each incubator was conditioned with fresh salts for a week in a temperature-controlled laboratory at $21 \pm 2^\circ\text{C}$ and relative humidity was monitored with a Fisherbrand Traceable thermometer and humidity monitor shown in Figure 4-22a.

The effects of moisture on accuracy and rate of response were examined by placing RFID MEMS sensors in enclosures and incubators at increasing relative humidity containing sodium nitrate (75% RH), potassium hydrogen sulphate (86% RH), potassium nitrate (93% RH) salt solutions and moisture (100% RH) for 15 minutes with measurements obtained every 5 seconds as shown in Figure 4-22 b. Similarly, RFID MEMS sensors were placed in incubators and enclosures at decreasing relative humidity containing moisture (100% RH), potassium nitrate (93% RH), potassium hydrogen sulphate (86% RH) and sodium nitrate (75% RH) salt solutions for 15 minutes with measurements obtained every 5 seconds.

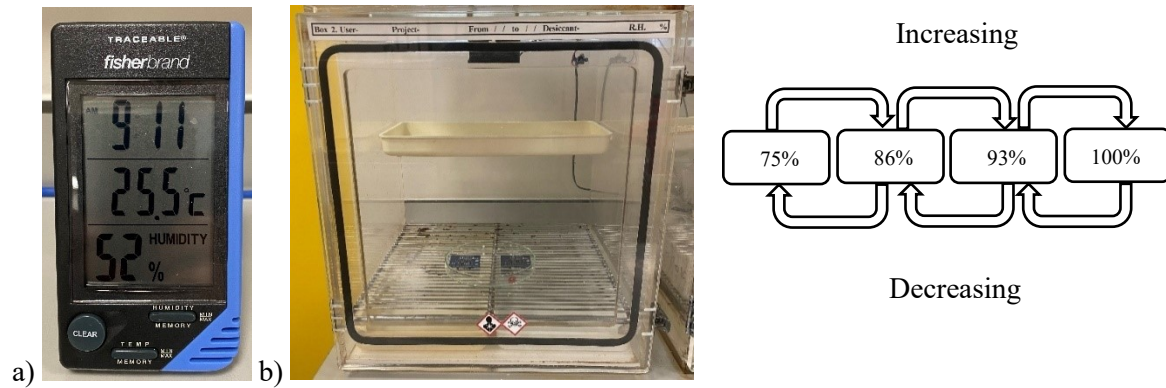


Figure 4-22 Conditioning incubators and enclosures a) with relative humidity examined by Fisherbrand Traceable thermometer and humidity monitor and b) for precision and accuracy testing with RFID MEMS sensors.

4.4.1 Results

4.4.1.1 Accuracy of MEMS

4.4.1.1.1 Increasing relative humidity

Figure 4-23 presents changes in relative humidity against time to understand the effects of moisture on the precision and accuracy of RFID MEMS sensors. Plots are separated into three different relative humidity ranges with Y-axes plotted on different scales. A maximum of 13 data points were plotted for each measurement to enhance clarity.

From the measurements obtained, it would seem that sensors have good accuracy with a maximum difference of $\pm 2\%$ once readings had reached an equilibrium. Similar trends were observed at 86% relative humidity with a difference of $\pm 1\%$ between measurements. This indicates that the RFID MEMS sensors tested were capable of providing reproducible results within the desired sensing range of 70 – 90% relative humidity. Nevertheless, results showed a higher deviation in measurements at higher relative humidity. This was evident with a maximum 6% deviation in measurements at 93% relative humidity. The variation in measurements could have resulted from high capillary forces and stiction (Patton, Cowan, and Zabinski 1999); where attractive forces have exceeded counteracting forces. In addition, condensation could have contributed to such effects. To improve the performance at higher

relative humidity environments, a 5 point calibration can be conducted with measurements obtained at relative humidity above 93 %. However, it is important to note the limitations and availability of saturated salts that are capable of providing relative humidity environments above 90 %. Furthermore, compared to manufacturer specifications the sensor was able to perform within the given accuracy of $\pm 3.5\%$ for the sensing ranging of 70 – 90 %.

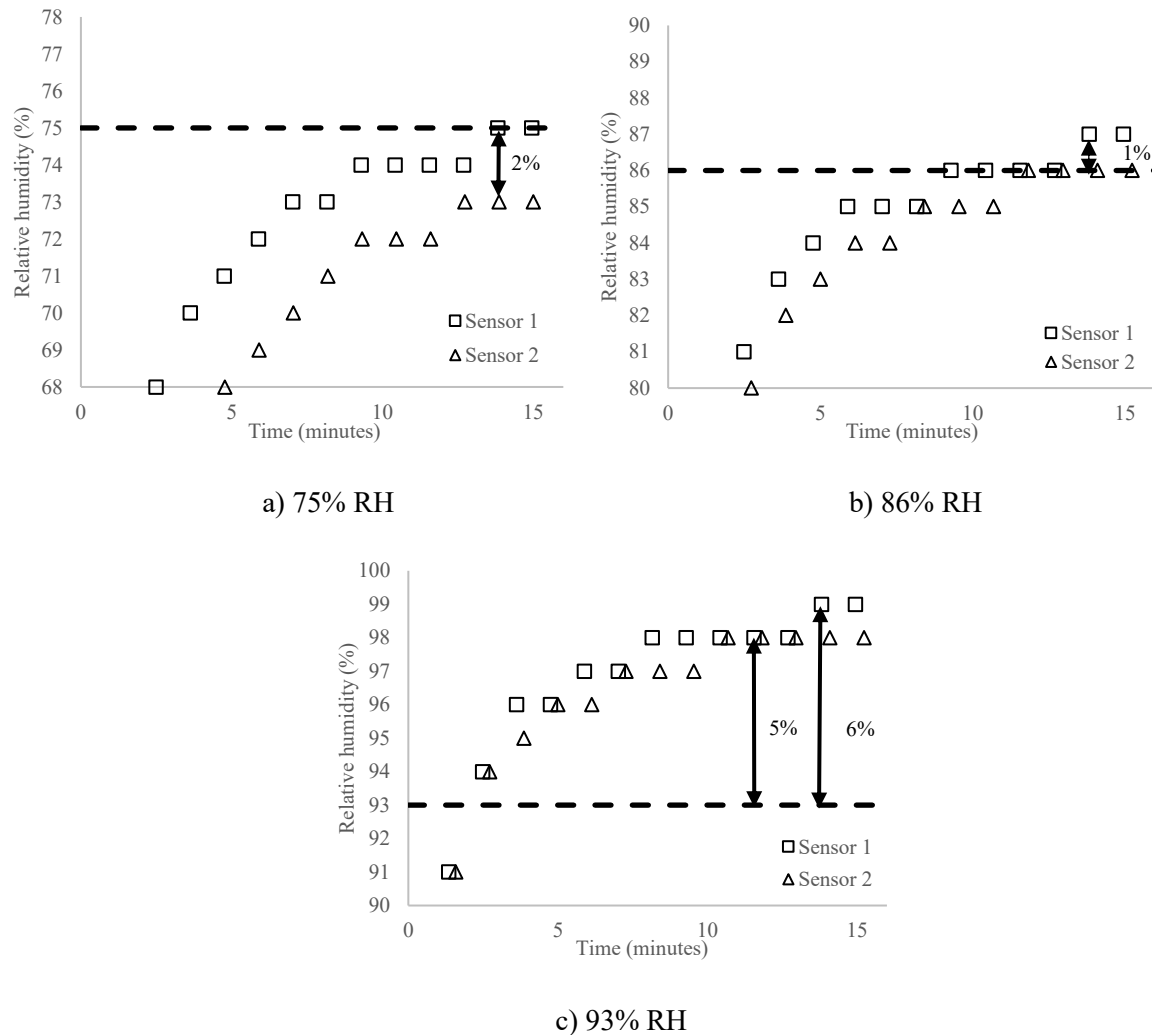
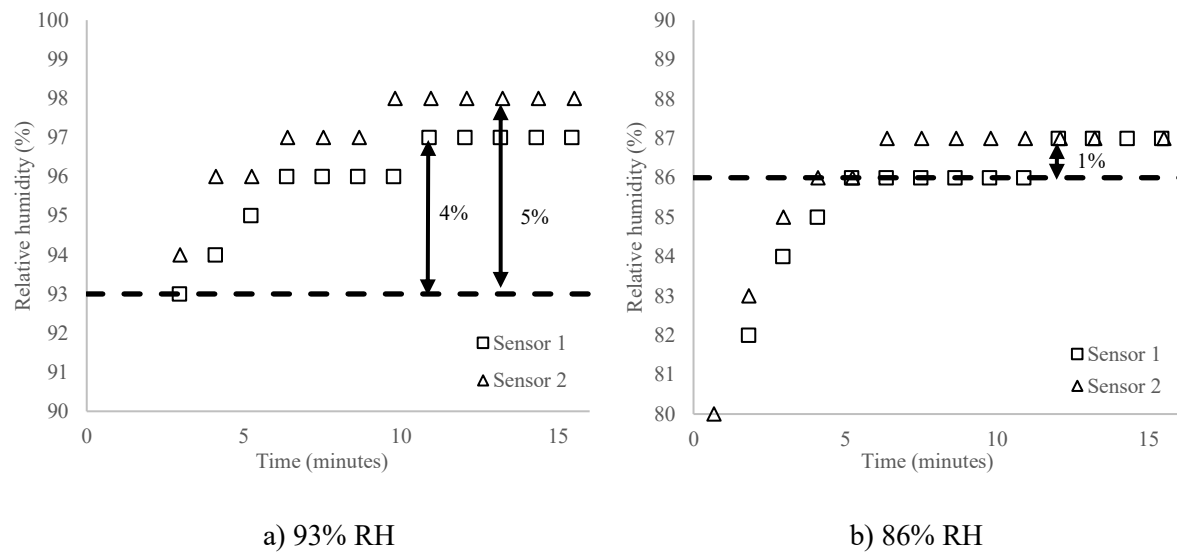


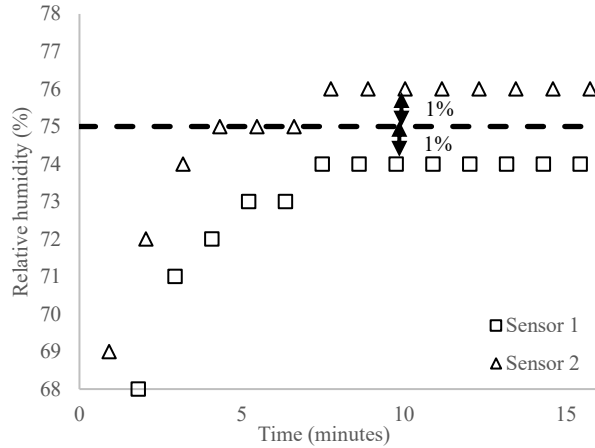
Figure 4-23 Evaluating the accuracy of RFID MEMS by placing sensors in incubators containing saturated salts in the order of sodium chloride (75% RH), potassium hydrogen sulphate (86% RH) and potassium nitrate (93% RH).

4.4.1.1.2 Decreasing relative humidity

Figure 4-24 plots the changes in relative humidity against time to understand the effects of moisture on the precision and accuracy of RFID MEMS sensor. Plots were separated into three different relative humidity ranges with Y-axes plotted on different scales. Datapoints were reduced to a maximum of 13 points per sensor measurement to enhance clarity.

Similarly, results obtained suggest that sensors have good accuracy with a maximum difference of $\pm 2\%$ between readings after reaching an equilibrium. This implies that reproducible results can be attained after exposing sensors from high to low relative humidity environments. Nonetheless, a deviation in measurements was observed at higher relative humidity. This can be observed with a maximum 5 % deviation in measurements at 93 % relative humidity.





c) 75% RH

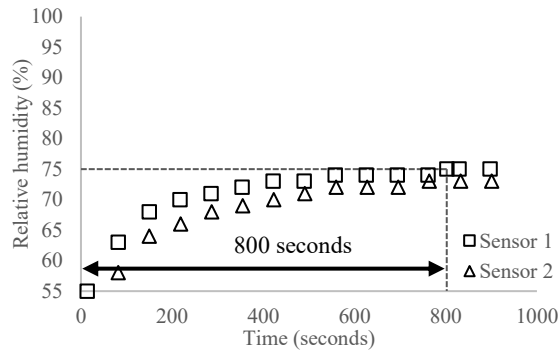
Figure 4-24 Evaluating the accuracy of RFID MEMS by placing sensors in incubators containing saturated salts in the order of potassium nitrate (93% RH), potassium hydrogen sulphate (86% RH) and sodium chloride (75% RH).

4.4.1.2 Rate of response

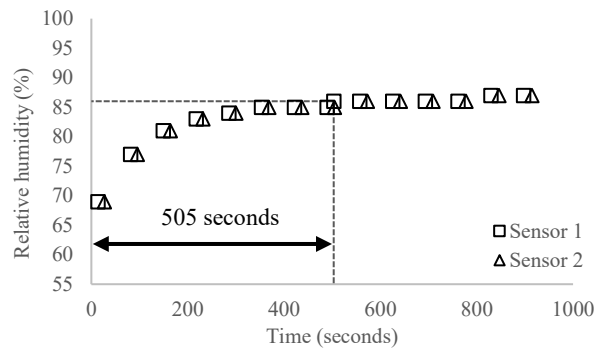
4.4.1.2.1 Increasing relative humidity

The changes in relative humidity against time are presented in Figure 4-25 to understand the effects of moisture on the rate of response. Plots were separated into four different relative humidity ranges with arrows to indicate the response time. It is established by the time required to detect and stabilize at the relative humidity of the surrounding environment from a controlled laboratory condition of 55 ± 5 % RH. In addition, a maximum of 16 data points were plotted for each measurement to enhance clarity.

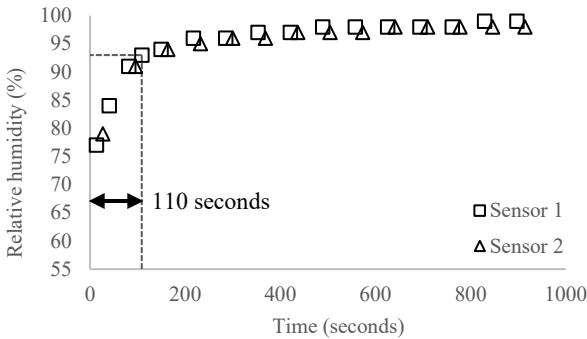
Measurements obtained suggest a lesser time was required for RFID MEMS sensors to stabilize at higher relative humidity. This can be observed with the detection times of 800 seconds for 75 % RH, 505 seconds for 86 % RH, 110 seconds for 93 % RH and 180 seconds for 100 % RH. The reduction in detection times can be influenced by an increase in the moisture content of the surrounding environment. Furthermore, it was interesting to note a longer time was required to detect 100% RH compared to 93% RH. This could be caused by moisture accumulating on the sensing element resulting in stiction affecting the performance and response.



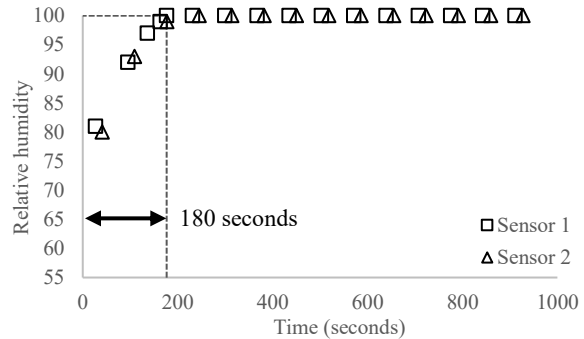
a) 75% RH



b) 86% RH



c) 93% RH



d) 100% RH

Figure 4-25 Examining the rate of response of RFID MEMS by placing sensors in incubators containing saturated salts in the order of sodium nitrate (75% RH), potassium hydrogen sulphate (86% RH), potassium nitrate (93% RH) salt solutions and moisture (100% RH).

4.4.1.2.2 Decreasing relative humidity

The changes in relative humidity against time are presented in Figure 4-26 to understand the effects of moisture on the rate of response. Plots were separated into four different relative humidity ranges with arrows to indicate the response time. It is established by the time required to detect and stabilize at the relative humidity of the surrounding environment from a controlled laboratory condition of 55 ± 5 % RH. Moreover, 15 data points were plotted for each measurement to enhance clarity.

Similar trends were observed with detection times of 435 seconds for 75 % RH, 300 seconds for 86 % RH and 180 seconds for 93 % RH. However, the sensor required 680 seconds to detect 100 % RH. This is because when introducing a sensor from a dry to a moist environment, the sensor will require time to detect and respond to moisture changes.

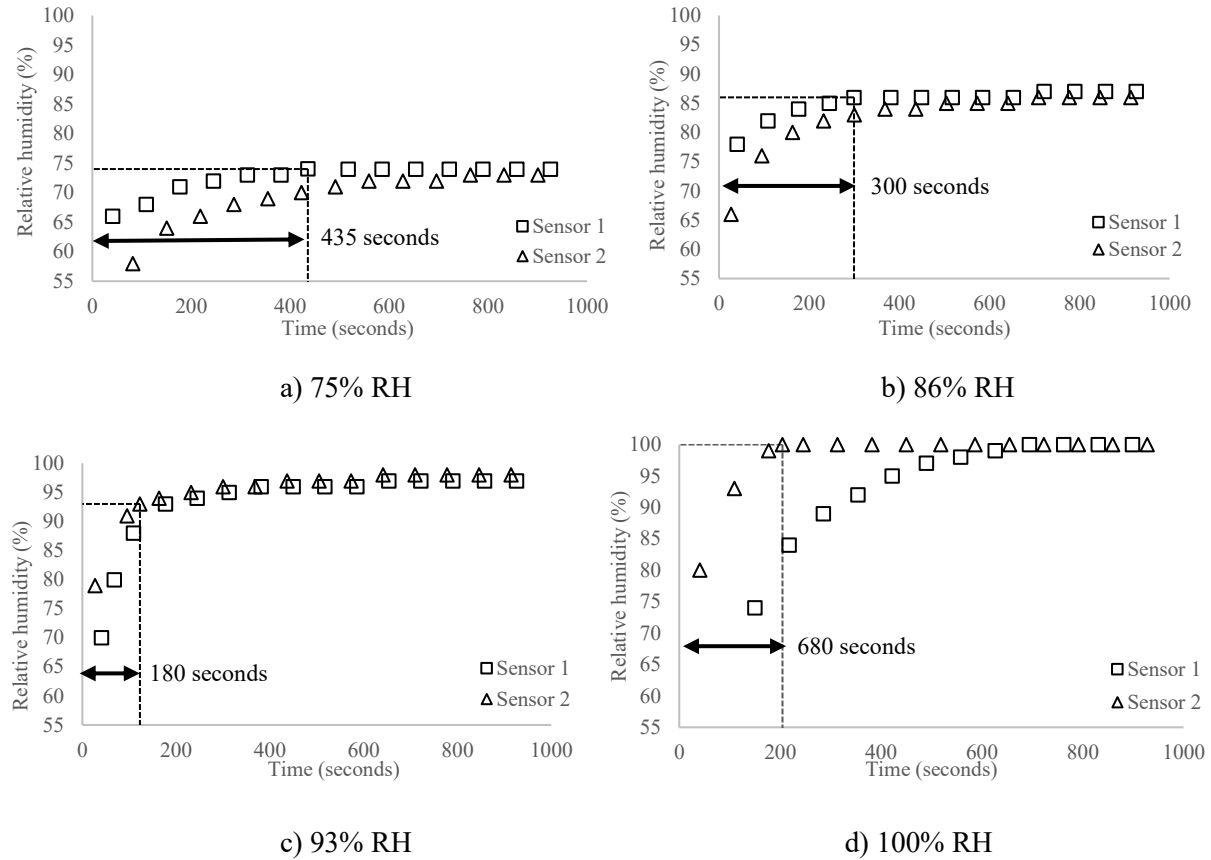


Figure 4-26 Examining the rate of response of RFID MEMS by placing sensors in incubators containing saturated salts in the order of moisture (100% RH), potassium nitrate (93% RH), potassium hydrogen sulphate (86% RH) and sodium nitrate (75% RH).

4.4.2 Discussion

4.4.2.1 Effects of moisture on accuracy, precision and response of RFID MEMS sensor

While developing a suitable method to monitor relative humidity at the steel-concrete interface, it is critical to study the accuracy of RFID MEMS sensors in controlled relative humidity environments. This

will reveal any limitations and provide opportunities for future developments. Results obtained suggest a deviation of measurements at a relative humidity of 93 %; however, precision was unaffected. This indicates that the polymer dielectric planar capacitor structure was functional in high relative humidity environments. Nonetheless, results from this study suggest that measurements can be affected by condensation and stiction shown in Figure 4-27. These observations are similar to experimental studies conducted with MEMS devices exposed to humid environments. Patton et al. (1999) observed a reduction in the performance of polysilicon MEMS cantilever motors at relative humidity above 70 % due to high capillary forces and stiction. Birleanu et al. (2017) measured a rise in pull-off forces between aluminium bridges and silicon substrate during an increase in relative humidity from 40 to 70 %.

To mitigate these issues, heating elements had been integrated into MEMS sensors (Dai 2007; Dai et al. 2007; Gu, Huang, and Qin 2004; Jorapur et al. 2015). Gu et al. (2004) fabricated a capacitive humidity sensor with a polysilicon heater based on 3 μm CMOS technology and observed a maximum hysteresis of 3% at 80% RH. Dai et al. (2007) developed a tungsten trioxide humidity sensor with a microheater and inverted amplifier circuit. Results from this study demonstrated that the sensor exhibited a small degree of hysteresis with a sensitivity of 4.5 mV/% RH at 60 °C and 4.8 mV/% RH at 75°C. Nevertheless, RFID MEMS sensors examined in this study demonstrated that hysteresis and inaccuracies still persist in environments above 93% relative humidity. On the other hand, the rate of response, sensitivity and precision was not affected during wetting and drying cycles.

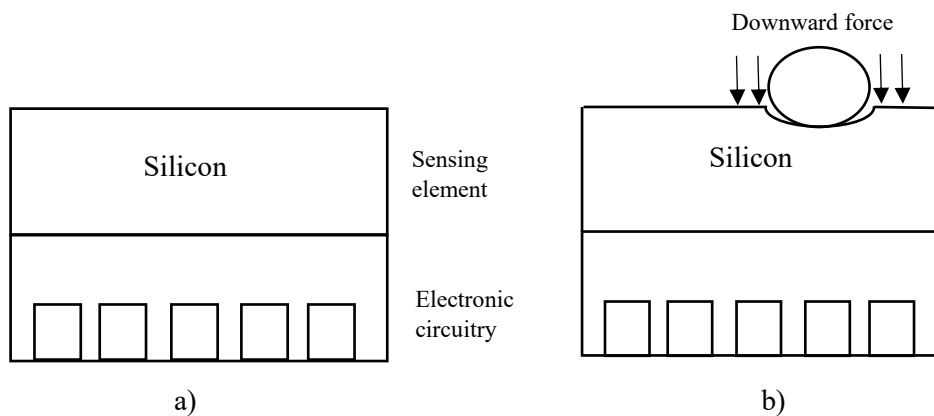


Figure 4-27 Components and structure of the sensing element a) without the influence and b) with the influence of stiction and moisture.

4.5 Wireless communication range

Four specimens with dimensions of $100 \times 100 \times 100$ mm were prepared. Concrete and mortar with the aggregate volume fraction of 73 and 58 % were dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Samples were cast in plastic moulds and compacted on a vibrating table until no significant release of air bubbles was observed. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of 20 ± 2 °C and relative humidity of 95 ± 5 % for 2 days. Once the desired curing age has been achieved, the cover depth was reduced to 50 mm with a Buehler Delta automatic abrasive cutter.

Communication distance was determined by placing a RIFD MEMS sensor in a polyethylene terephthalate enclosure with the antenna surface in direct contact with the specimen shown in Figure 4-28 a. Subsequently, electromagnetic waves were continuously emitted from the ST25R3911B-DISCO RFID reader with the continuous scan mode allocated on the ST25PC-NFC software. The reader was shifted at a distance of 5mm increments until the RFID MEMS sensor was undetectable and the maximum communication distance was measured with a vernier calliper. In addition, specimens were submerged in tap water for 5, 15, 30 and 60 minutes to study the impact of moisture on wireless communication range. Further tests included placing the RFID MEMS sensor at 15°, 30°, 45°, 50° and 60° away from the plane parallel to the RFID reader to investigate the effect of directional placement on the communication range shown in Figure 4-28 b.

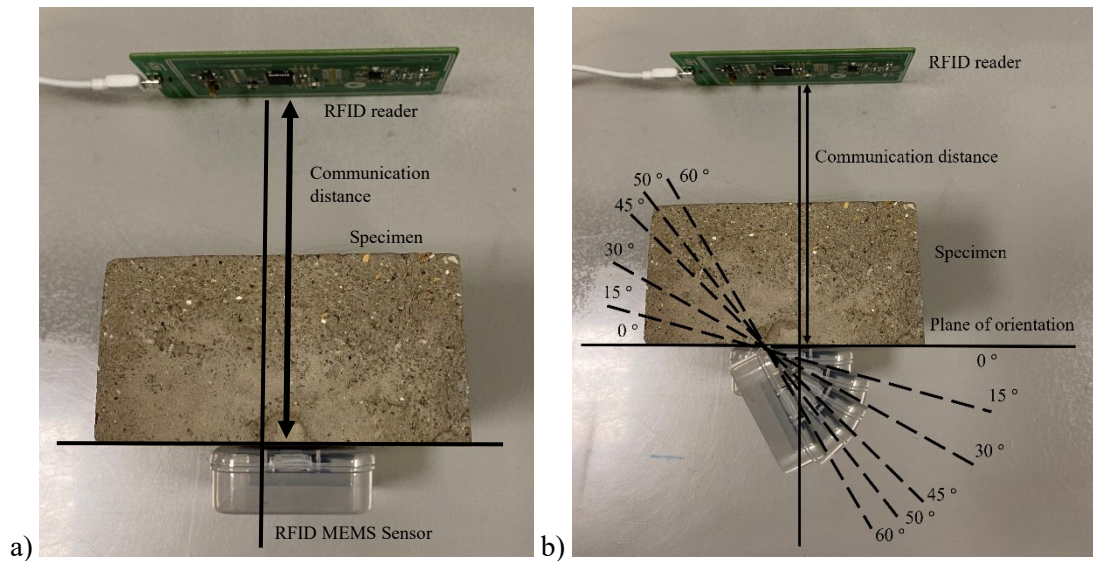


Figure 4-28 Test method to determine the maximum communication distance by placing RFID MEMS sensor a) below 50 and 100mm concrete and mortar specimens and b) at different orientations.

4.5.1 Results

4.5.1.1 Effects on wireless communication range

4.5.1.1.1 Influence of aggregate content and degree of saturation

Figure 4-29 presents the communication range against submergence time to understand the effects of moisture and aggregate content. An additional plot was included to better quantify the percentage reduction in communication range against submergence time. Near identical communication distances were measured with replicate samples; thus, average values were presented for clarity.

From the measurements obtained, a reduction in communication range of 16 mm was observed for mortar with a 50 mm cover compared to 11.5 mm for concrete with a 50 mm cover after submerging in tap water for 60 minutes. By interpreting communication range as a percentage reduction, it was revealed that mortar had a 13.7 % reduction in comparison to concrete with a 9.9 % reduction. These observations are expected because water to cement ratio, aggregate volume fraction and aggregate size would influence

sorptivity, total absorption and accessible porosity. Hence, altering the overall dielectric properties of specimens. Moreover, it was interesting to note that an increase in cover depth has not affected the overall communication distance between the sensor and reader. Both concrete and mortar specimens with 50 and 100 mm cover depth had a maximum communication distance of 115 ± 5 mm.

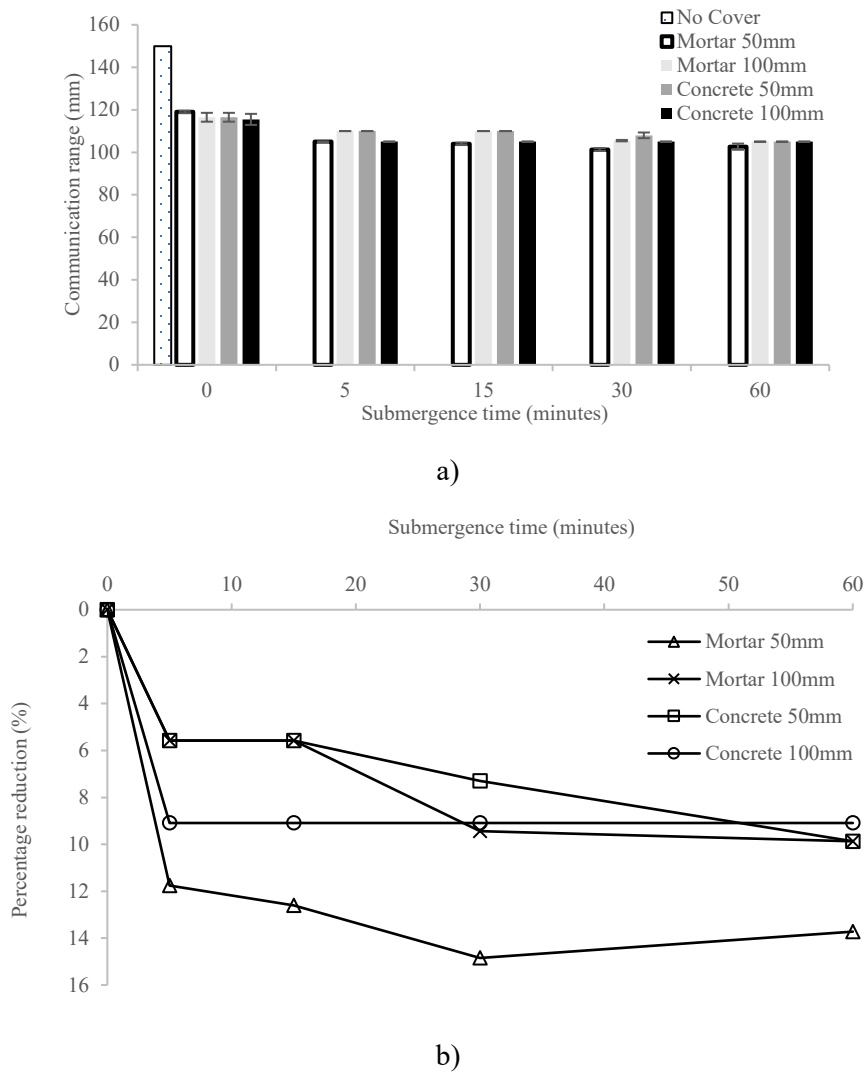
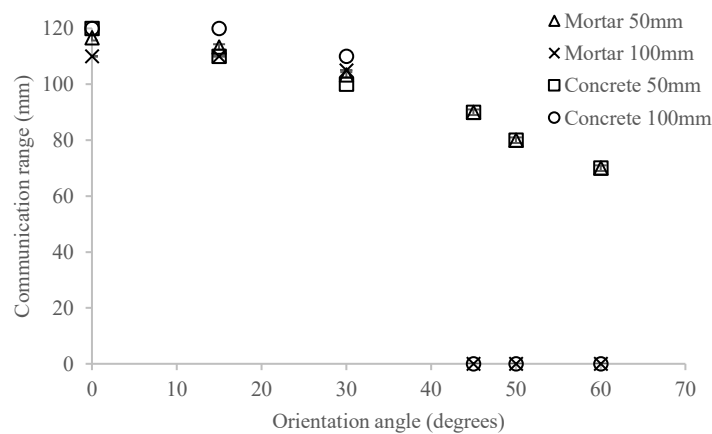


Figure 4-29 Change in maximum communication range measured after submerging specimens in tap water.

4.5.1.1.2 Directional placement

To study the impact of directional placement between RFID MEMS sensor and reader communication range was measured and shown in Figure 4-30. A second plot of percentage reduction in communication range against orientation angle was presented for better quantification and clarification. Replicate samples produced near identical communication distances; thus, average values were presented for clarity.

From the results presented, it can be observed that near identical measurements were obtained for mortar and concrete specimens with similar cover depths. By tilting the RFID MEMS sensor 15° away from the horizontal plane both mortar and concrete specimens with 50mm cover depth had a maximum communication distance of 110 mm. Likewise, at 30° mortar and concrete specimens with 50 cover depth had a communication distance of 100 mm. This implies that an increase in aggregate volume fraction and size has not affected communication range. On the other hand, RFID MEMS sensors were undetectable for both mortar and concrete specimens with 100mm cover depth at 45° orientation.



a)

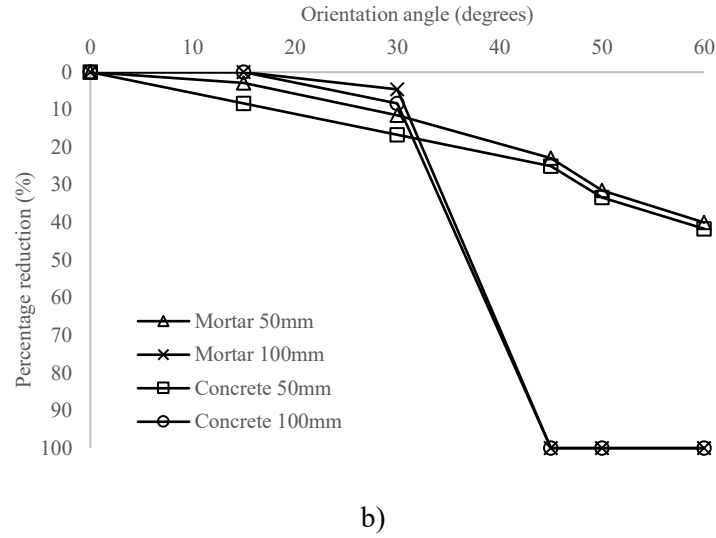


Figure 4-30 Change in maximum communication range measured by tilting RFID MEMS sensor away from RFID reader.

4.6 Transmitted power analysis

Twelve specimens with dimensions of $50 \times 50 \times 25$ mm were prepared. Mortar was dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Cement paste was batched in a Hobalt mixer. Samples were cast in $50 \times 50 \times 50$ mm steel moulds and compacted on a vibrating table until no significant release of air bubbles was observed. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $95 \pm 5\%$ and cured for 7 days. Once desired curing ages have been achieved, the cover depth was reduced to 25mm with Isomet 1000 precision cutter (Figure 4-31). Subsequently, selected specimens were conditioned in 50 degree oven, saturated sodium chloride salt solution and submerged in tap water for four weeks till a mass change smaller than 0.01% was obtained.

Transmitted power received by the RFID MEMS sensor was monitored by soldering 2.5mm diameter copper wires onto the R6 resistor and diode (Figure 4-32 b). After soldering, wires were attached to a Solartron 7050 digital multimeter with crocodile clips and electromagnetic waves were continuously emitted from an ST25R3911B-DISCO RFID reader. The minimum and maximum communication

distance was determined by shifting the RFID reader at a distance of 20mm increments till the RFID MEMS sensor was undetectable. To ensure that measurements were not affected by environmental factors, experiments were conducted in a temperature-controlled laboratory at $21 \pm 2^\circ\text{C}$ and $55 \pm 5\%$ RH.

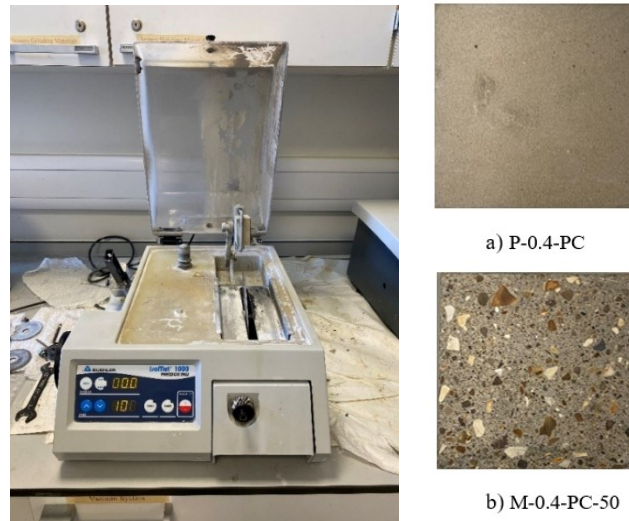


Figure 4-31 Sectioning mortar and cement paste specimens with Isomet 1000 precision cutter of mortar.

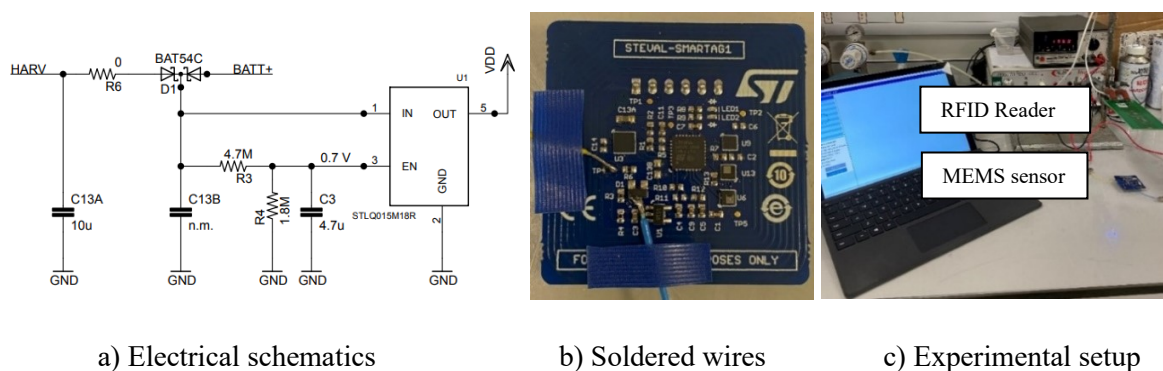


Figure 4-32 Electrical schematics of ST STEVAL SMARTAG 1 with 2.5mm diameter wires soldered onto the R6 resistor and diode attached to a Solartron 7050 digital multimeter to monitor peak transmitted power.

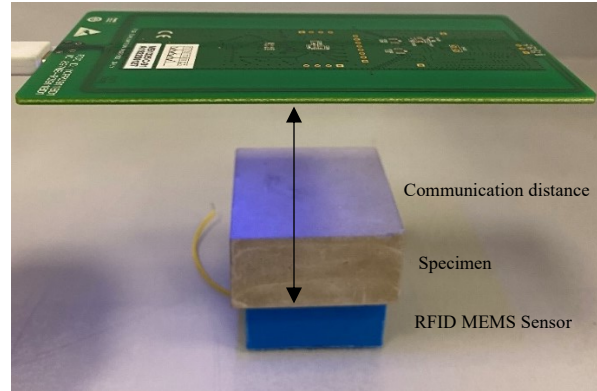


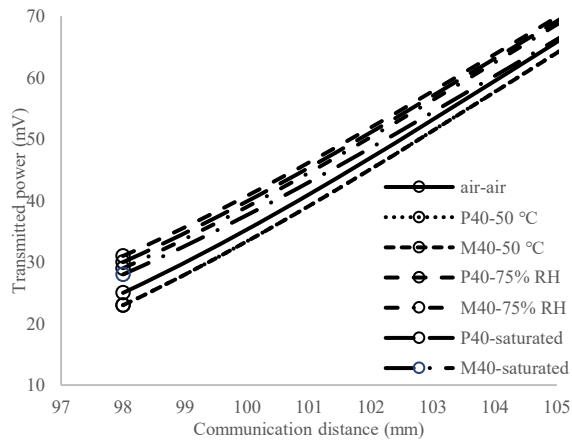
Figure 4-33 Placement of mortar and cement paste specimens above RFID MEMS sensor to determine the maximum transmission power received.

4.6.1 Results

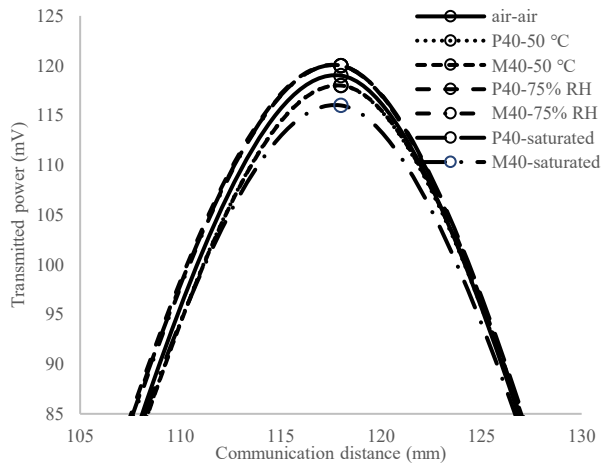
4.6.1.1 Effects on transmitted power

Figures 4-34 and 4-35 plot changes in transmitted power against communication distance to study the effects of moisture and aggregate content. To enhance clarity, plots were separated into three different communication distances with Y-axes and X-axes plotted on different scales. Near identical measurements were obtained with replicate samples; hence, the average value was presented for clarity.

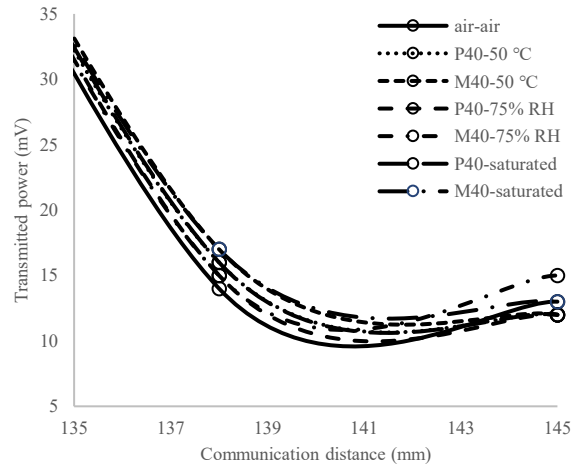
The overall trend suggests that near identical transmitted power was measured for air-air and specimens placed above the RFID MEMS sensor. This indicates that cementitious material, aggregates and moisture do not have a huge influence on transmitted power for wireless sensor technologies operating at 13.56 MHz frequency. Nonetheless, it was interesting to observe that both cement paste and mortar specimens had enhanced transmitted power received at communication distances of 97-105 mm and 135-145 mm. This can be explained based on the dielectric nature of cementitious materials, where electromagnetic waves can reflect and transmit through pores and aggregates. Moreover, a parabolic behaviour was observed from the measurements obtained (Figure 4-35). This is because the sensor was able to receive a maximum amount of electromagnetic fields per unit area at an optimum distance away from the RFID MEMS sensor.



a) 97-105 mm



b) 105-130 mm



c) 135-145 mm

Figure 4-34 Peak transmitted power received by RFID MEMS sensor measured at a) 97-105 mm, b) 105-130 mm and c) 135-145 mm communication distances.

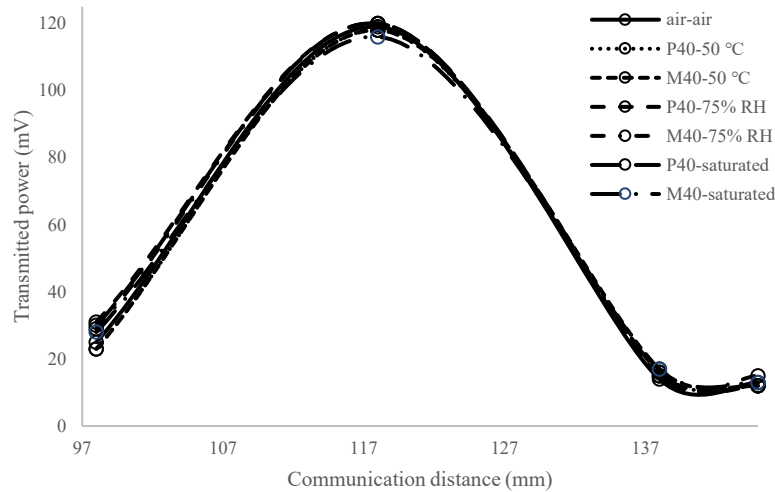


Figure 4-35 The influence of moisture and aggregate content on transmitted power at 13.56 MHz frequency.

4.6.2 Discussion

4.6.2.1 Influence of directional placement, moisture and aggregate content on wireless communication and transmitted power

With known measurements of transmitted power and air-air communication distance, the effects of cover depth, water to cement ratio and aggregate volume can be established. Results from this study showed that an increase in cover depth, water to cement ratio and aggregate volume have not affected the communication distance between the reader and sensor. These findings are in agreement with radio frequency measurements obtained for cementitious materials (Daly, Melia, and Baldwin 2010). At frequencies below 100 MHz, attenuation is similar to free space; hence, communication distance and signal strength will not be considerably affected. Similarly, transmitted power measurements suggest that wireless communication was unaffected by cementitious barrier materials. These observations are similar to studies with RFID tags embedded in cementitious material (Bouzaffour et al. 2021; Donno et al. 2013; Zhou et al. 2016). Zhou et al. (2016) investigated the return loss performance of 915 MHz RFID tags with saturated concrete and air and observed similar bandwidths and transmission losses. Donno et al. (2013)

studied the influence of barrier materials on reader-tag distance at 830-930 MHz frequency band and discovered similar communication distances and peak sensitivity for wood and concrete materials. Similarly, Bolton et al. (2017) observed identical communication distances with and without barrier materials consisting of metal, cardboard and air. Moreover, the communication range was significantly reduced by rotating the RFID MEMS sensor away from the RFID reader. This can be attributed to a polarization mismatch between the transmitting and receiving antenna. These findings are in agreement with experimental studies conducted with RFID sensors and transponders operating at the radio frequency range of 13.56 MHz (H. M. Lu et al. 2007; Sakonkanapong and Phongcharoenpanich 2020).

4.7 Enclosure design

To study the impact of enclosure design on relative humidity measurements, RFID MEMS sensors without enclosures were placed above two 5×5 mm plastic rods in containers containing shallow water at a depth of 4 mm (Figure 4-36). Subsequently, RFID MEMS sensors were embedded in enclosures and placed above two 5×5 mm plastic rods in containers containing shallow water at a depth ranging from 1-4 mm (Figure 4-37). Measurements were obtained at 5 second intervals for 24 hours. Throughout the test, containers were covered with a lid and care was taken to ensure that the water level was maintained.



Figure 4-36 Directly exposing RFID MEMS sensors to 100% RH humidity.

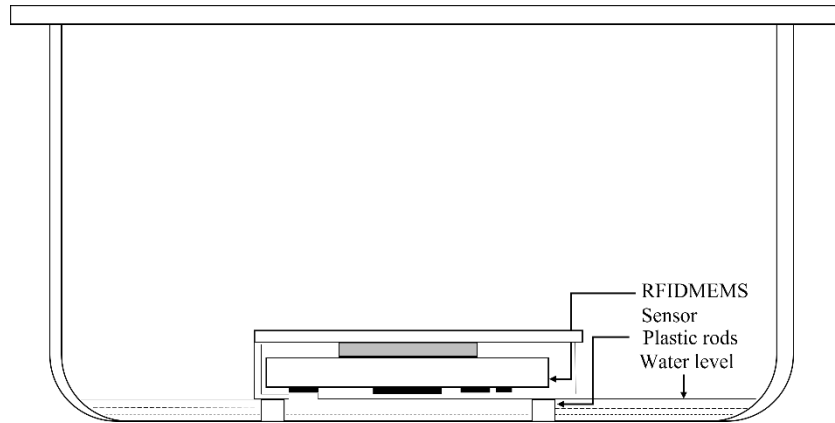


Figure 4-37 Directly exposing RFID MEMS sensors with an enclosure to 100% RH humidity.

4.7.1 Results

4.7.1.1 Influence of enclosure design

The rate of increase in relative humidity against elapsed time was plotted in Figure 4-38 to examine the influence of enclosure design. To develop a clear analysis, a maximum of 38 data points were plotted for each measurement.

Measurements obtained showed a slower diffusion of moisture into enclosures compared to direct exposure. At identical water depths of 4 mm, a measurement of 100 % RH was obtained at approximately 9 minutes for RFID MEMS sensor without enclosure compared and 127 minutes for RFID MEMS sensor with enclosure. Furthermore, by reducing the water depths to 1 mm and 2 mm the measured relative humidity at 127 minutes was 91% and 85%. This indicates that the abundance of dead space in enclosures will influence the time required for the internal volume of an enclosure to stabilize at a moisture state even though an initial rise in relative humidity was observed for all sensors within a few minutes of exposure. Additionally, lower relative humidity measurements were obtained at shallower water depths as a result of longer diffusion times.

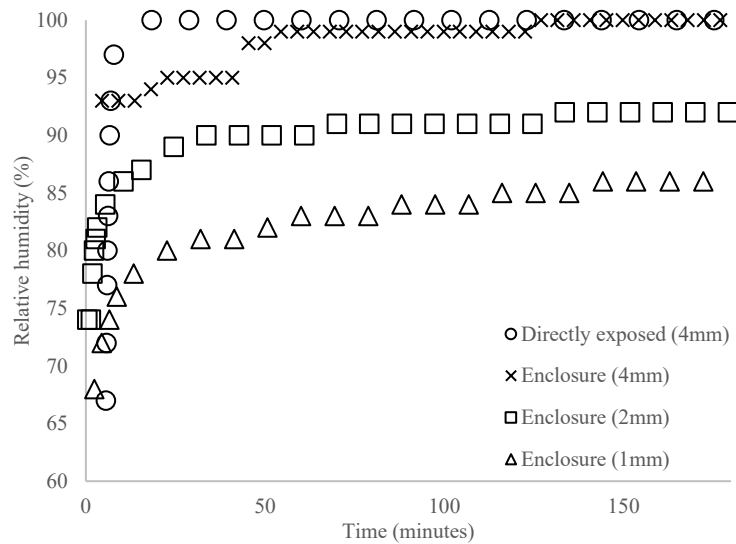


Figure 4-38 Monitoring changes of relative humidity for RFID MEMS sensors with and without enclosures.

4.7.1.2 Discussion

By measuring changes in relative humidity over a period of time, the influence of enclosure design on measurements can be examined. Results from this study demonstrated that sensors were able to detect changes in relative humidity but a longer time was required for enclosures to stabilize at a desired moisture state (Figure 4-38). This can indicate the diffusion of saturated air between sensor components and battery compartments despite the fact that enclosure designs were optimized for this study (Figure 4-39). Therefore, this can affect diffusion times monitored by RFID MEMS sensors with identical methods adopted in Section 4.8 and Chapter 6. Although, the application of resin material was considered to reduce the amount of dead space this will induce stress and strains on sensor components and can potentially affect the accuracy and reliability of measurements (4.3.2.3).

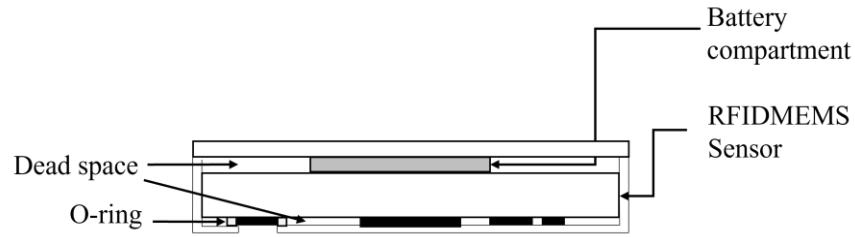


Figure 4-39 RFID MEMS sensor embedded in 3D printed enclosure with an abundance of space around sensor components and battery compartment.

4.8 Monitoring relative humidity

Fourteen specimens of 100 mm diameter and thickness of 45, 60 and 85 mm were prepared. High and low-strength concrete with an aggregate volume fraction of 70 % were dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Before casting, 3D-printed enclosures with 8mm diameter steel bars were placed in plastic moulds (Figure 4-40 a). Samples were compacted in two to three layers on a vibrating table until no significant release of air bubbles was observed. To simulate improper compaction, selected high-strength concrete samples were compacted with a single layer (Figure 4-41). Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $95 \pm 5\%$ and cured for 3, 14 and 28 days. Subsequently, the sides of specimens were coated with epoxy and sealed with adhesive tape to ensure one-sided absorption and desorption shown in Figure 4-40 b. To monitor changes in relative humidity at the steel interface, specimens were conditioned in 50°C oven for four weeks. Subsequently, samples were conditioned in incubators containing saturated ammonium nitrate and potassium nitrate till a mass change smaller than 0.01% was obtained.

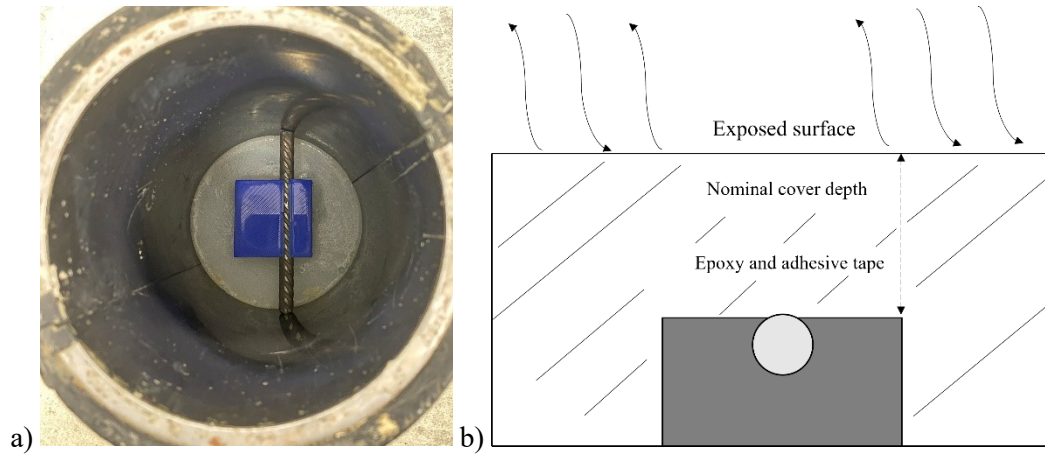


Figure 4-40 Embedment of 3D printed enclosures a) in 100 mm diameter plastic moulds and b) schematic design of developed method in concrete specimens.



Figure 4-41 High strength concrete 14 days cured specimens with a) improper and b) proper compaction.

4.8.1 Results

4.8.1.1 Rate of decrease in relative humidity measurements

The rate of decrease in relative humidity against elapsed time was plotted in Figure 4-42 to examine the influence of curing age, water to cement ratio and improper compaction. To develop a clear analysis, results with identical curing ages were sorted into groups.

Data obtained show a tendency for moisture to retain in specimens with a greater curing age and lower water to cement ratio. At approximately 283 hours, internal relative humidity measurements for LSC 3D, HSC 3D, LSC 14D and HSC 14D were 25 %, 48 %, 37 % and 54 % respectively. These observations are plausible because specimens have a lower abundance of empty pores (Nokken and Hooton 2002). Thus, the overall rate of desorption can decrease and require a longer time to detect changes in relative humidity. Moreover, there was a significant decrease in relative humidity for specimen without sufficient compaction with a measurement of 25% RH at 283 hours. This is resulted in insufficient adhesion between aggregates and cement paste. Additionally, there was an anomaly in relative humidity measurements for low and high-strength concrete samples at 33-133 hours as shown in Figure 4-42 b. This might have resulted from condensation when removing specimens from a 50°C oven to a temperature-controlled laboratory at $21 \pm 2^\circ\text{C}$ to obtain measurements.

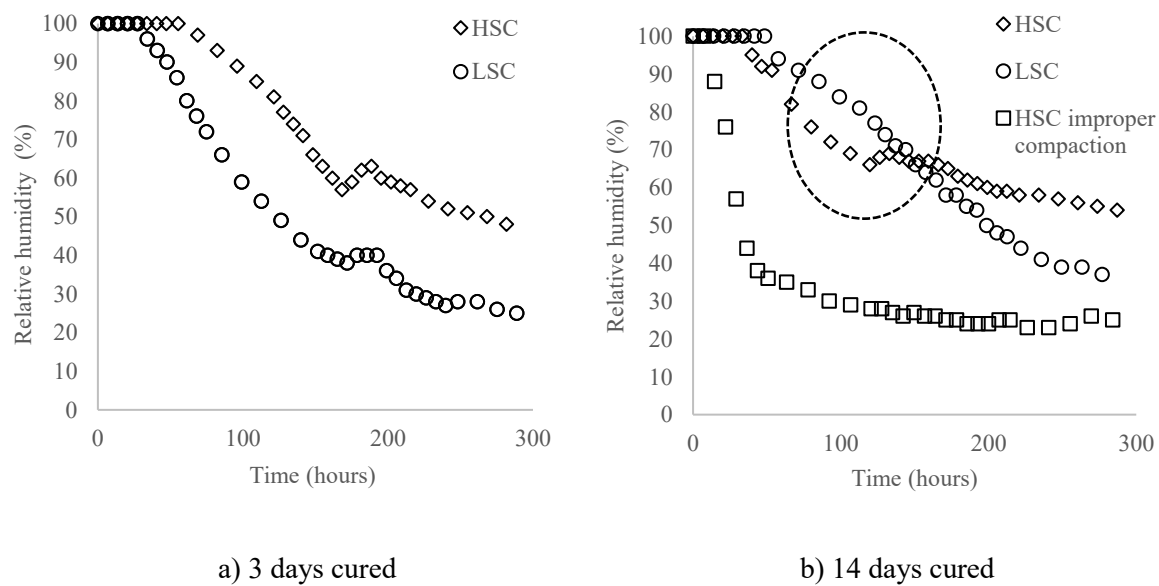


Figure 4-42 Monitoring changes of internal relative humidity at the steel-concrete interface with RFID MEMS sensors in low and high-strength concrete specimens with 10mm nominal cover depth during oven drying at 50°C.

4.8.1.2 Rate of increase in relative humidity measurements

Figure 4-43 presents the changes in relative humidity against elapsed time to investigate the effects of curing age, water to cement ratio, nominal cover depth and improper compaction. Plots with identical nominal cover depth and conditioning environment were allocated into groups for clearer analysis.

The overall trend suggests a greater increase in relative humidity for specimens with higher water to cement ratio and lower curing age. Measurements acquired at 833 hours indicated an internal relative humidity of 67 %, 61 %, 59 % and 57 % for LSC 3D, HSC 3D, LSC 14D and HSC 14D respectively. These findings are expected because samples have a higher abundance of empty pores (Nokken and Hooton 2002). Hence, the overall rate of absorption will increase and require a shorter time to notice changes in relative humidity. In addition, it was interesting to note a slower increase in relative humidity for specimens with improper compaction. This is because the diffusivity of moisture is restricted from an insufficient adhesion between aggregates and cement paste. Similarly, relative humidity increases at a slower rate for samples with a greater curing age and nominal cover depth (Figure 4-43 b). This can be resulted from a slower diffusion of moisture through finer pores and thicker cover.

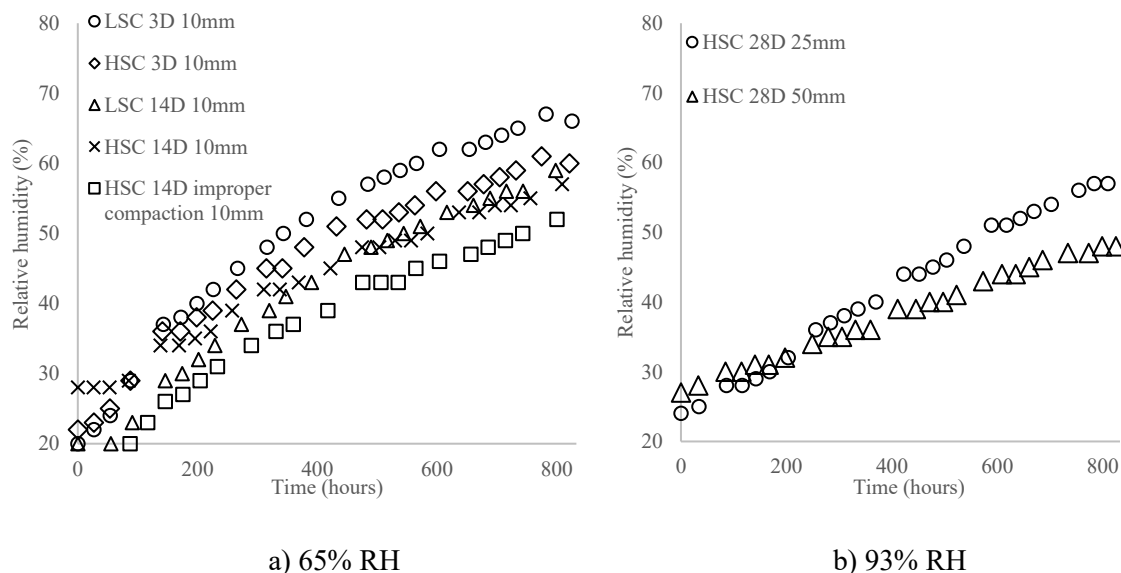


Figure 4-43 Monitoring changes of internal relative humidity at the steel-concrete interface with RFID MEMS sensors in low and high-strength concrete specimens with 10, 25 and 50 mm nominal cover depth.

4.8.2 Discussion

With relative humidity measurements obtained over a period of time, the rate of saturation at the steel-concrete interface can be revealed. Results from this study demonstrated a slower rate of increase and decrease in relative humidity for specimens with a greater curing age, lower water to cement ratio and increase in nominal cover depth shown in Figures 4-42 and 4-43. This is because moisture will have to diffuse through finer pores and travel longer distances. These results are aligned with numerical and experimental studies conducted (K. Li, Li, and Chen 2009; Nguyen et al. 2017). Nguyen et al. (2017) embedded a linear array four-point probe into concrete specimens to measure moisture content in the concrete cover. It was discovered that resistivity was lower at 20mm depth compared to 40mm depth which suggests a decrease in water content for specimens with thicker cover depth. From analytical approaches, Li et al. (2009) identified an increase in humidity gradient with an increased cover depth and concrete porosity.

Additionally, results from this study clearly illustrate a significant increase and decrease in relative humidity for specimens without proper compaction during wetting and drying. This indicates that the transport of moisture was directly impacted by the connectivity of voids from an insufficient adhesion of cement paste and aggregates at the steel-concrete interface (Samaha and Hover 1992).

4.9 Conclusions

The suitability of embedding MEMS sensors at the steel-concrete interface to monitor relative humidity changes was examined. This includes prototyping and customizing enclosures to provide sufficient protection, monitoring communication range and transmitted power with cement paste, mortar and concrete specimens, and examining relative humidity changes at the steel-concrete interface with low and high-strength concrete specimens placed at 50 °C, 65% and 93% RH environments. The study was an attempt to develop a novel technique to monitor relative humidity at the steel-concrete interface, with the overall aim of better understanding the suitability of embedding RFID MEMS sensors.

The main findings are as follows:

- a) RFID MEMS sensors placed in incubators containing saturated sodium nitrate (70 % RH), potassium hydrogen sulphate (86 % RH), potassium nitrate (93 % RH) salt solutions and moisture (100 % RH) suggest that precise measurements can be obtained with a maximum difference of ± 2 %.
- b) When placing RFID MEMS sensors in environments above 93% RH it was found that there was a reduction in accuracy with a deviation in results. This can be caused by moisture present on the sensing element due to condensation and stiction. To mitigate these issues, integrated heating elements can be operated to improve performance and accuracy.
- c) A reduction in communication distance was observed when tilting the RFID MEMS sensor away from the RFID reader as a result of a polarization mismatch between the transmitting and receiving antenna.
- d) An increase in moisture and aggregate content had not affected received transmitted power and communication range.
- e) A slower rate of increase and decrease in relative humidity was monitored for specimens with a greater curing age, lower water to cement ratio and thicker nominal cover depth. This is because the resistance to moisture transport is greater in these cases.
- f) Although the designed enclosure was able to provide direct relative humidity measurements at the steel-concrete interface. A significant amount of time was required for the internal volume to stabilize at a moisture state.

Chapter 5 Examining the reliability and performance of RFID-MEMS sensors exposed to cementitious materials and simulated marine environments

This chapter reports on the reliability and performance of RFID-MEMS sensors. The impact of proper enclosure design on the contamination of the MEMS sensor will also be discussed. New methods were developed to study the influence of cementitious materials, sodium chloride and moisture. Tests involved seven months of embedment in concrete, one year of cyclic wetting and drying, and 2 months of exposure to 3% wt. NaCl salt solution and 100% RH environments. It was shown for the first time RFID MEMS sensors were able to perform reliably after embedding in high and low-strength concrete and exposure to moisture; however, performance was affected after exposure to 3% wt. NaCl salt solutions for 63 days. Further tests were conducted to examine the degradation of sensor components with SEM-BSE and EDS. This include discussions on the effects of particulate contamination, moisture and chlorides on silicon thin films and electronic components.

5.1 Introduction

During in-situ monitoring, MEMS sensors will inevitably be exposed to cementitious materials, corrosive and humid environments. Changes in moisture content and alkalinity of the measured environment will reduce tensile strength and increase frictional wear for silicon substrates (Birleanu et al. 2017; Broas et al. 2015; D. Tanner et al. 2000; Yamaji et al. 2007). Degradation of sensor components will cause readings to be misinterpreted and result in inaccurate assessments of corrosion risk. Therefore, the capability to analyze and evaluate the reliability of sensors is highly beneficial for many research and field applications.

Numerous methods have been implemented to improve the accuracy and reliability of MEMS sensors. This includes the integration of heating elements, application of protective coatings and usage of alternative sensing materials (Brazzle et al. 2003; Choudhary, Kharat, and Kaur 2011; Dai et al. 2007). Regardless of the potential enhancements, the applicability of these techniques for in-situ monitoring is

unknown. This is resulted from the lack of studies for characterising the degradation and performance of RFID MEMS sensors exposed to cementitious materials and marine environments, which supports the need for this work.

This study aims to investigate the effects of cementitious materials and marine environments on sensor components. RFID MEMS sensors were embedded in low and high-strength concrete and directly exposed to saturated sodium chloride salt solutions and moisture. Surface morphology was examined with SEM and EDS, and related to relative humidity measurements to explain changes in performance.

5.2 Preliminary studies

The effect of packaging techniques on particulate contamination was examined by embedding RFID MEMS sensors in commercially available polyethylene terephthalate and customized 3D-printed PLA enclosures with the schematics shown in Sections 4.3.1 and 4.3.4. Subsequently, specimens were placed under an Olympus BX51 stereomicroscope with the equipment and setup shown in Section 3.3.2

5.2.1 Materials and mix proportions

Concrete with a water/cement ratio (w/c) of 0.6 and an aggregate volume fraction of 73% was prepared. Cement I 52.5 R was used with the chemical and physical properties specified in Section 3.1.1. Mixes were designed with the proportions shown in Table 5-1.

Table 5-1 Mix proportions.

<i>Mix design</i>	<i>w/c ratio</i>	<i>Water (kg/m³)</i>	<i>Sand (kg/m³) <5mm</i>	<i>Coarse aggregate (kg/m³) <10mm</i>	<i>Cement (kg/m³)</i>	<i>Aggregate vol fraction: %</i>
C-0.6-PC-73	0.6	175.6	750.3	1125.4	292.7	73

5.2.2 Samples

5.2.2.1 Commercially purchased enclosure

Two specimens with the dimensions of $100 \times 100 \times 100$ mm were prepared. Concrete was dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Samples were cast in plastic moulds and compacted on a vibrating table until no significant release of air bubbles was observed with polyethylene terephthalate enclosures held in position at 10mm depth from the surface. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $95 \pm 5\%$ for 28 days.

5.2.2.2 Customized PLA enclosure

Two specimens of 100 mm diameter and thickness 85 mm of were prepared. Concrete was dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Before casting, 3D printed enclosures with 8mm diameter steel bars were placed in plastic moulds. Samples were compacted in two to three layers on a vibrating table until no significant release of air bubbles was observed. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $95 \pm 5\%$ and cured for 28 days.

5.2.2.3 Olympus BX51 stereomicroscopy

The surface morphology of MEMS sensors was examined after 28 days of embedment in concrete. This was done by retrieving the specimens from enclosures and removing any excess dust particulates with the application of compressed gas with an RS PRO air duster. Subsequently, specimens were placed under an Olympus BX51 stereomicroscope with the equipment and setup shown in Section 3.3.2. Images were captured at 5 times magnification with a working distance of 12 mm.

5.2.3 Results and discussion

Figure 5-1 presents surface morphology images of MEMS sensors before and after embedding in concrete for 28 days. From a close inspection, slight manufacturing defects were found on MEMS sensors (Figure 5-1 a). These observations are expected due to adhesion and degradation issues from mass and batch manufacturing of silicon and polymers (Y. Li 2018). During hydrofluoric acid etching of silicon dioxide on a silicon wafer, organic and inorganic materials can be deposited on sensors. Moreover, it was interesting to observe signs of contamination and degradation after embedding MEMS sensors in commercially purchased polyethylene terephthalate enclosures. This indicates that the application of waterproof polyester materials was unfeasible. In comparison, no signs of contamination or degradation were observed for MEMS sensors embedded in 3D printed enclosures (Figure 5-1 c).

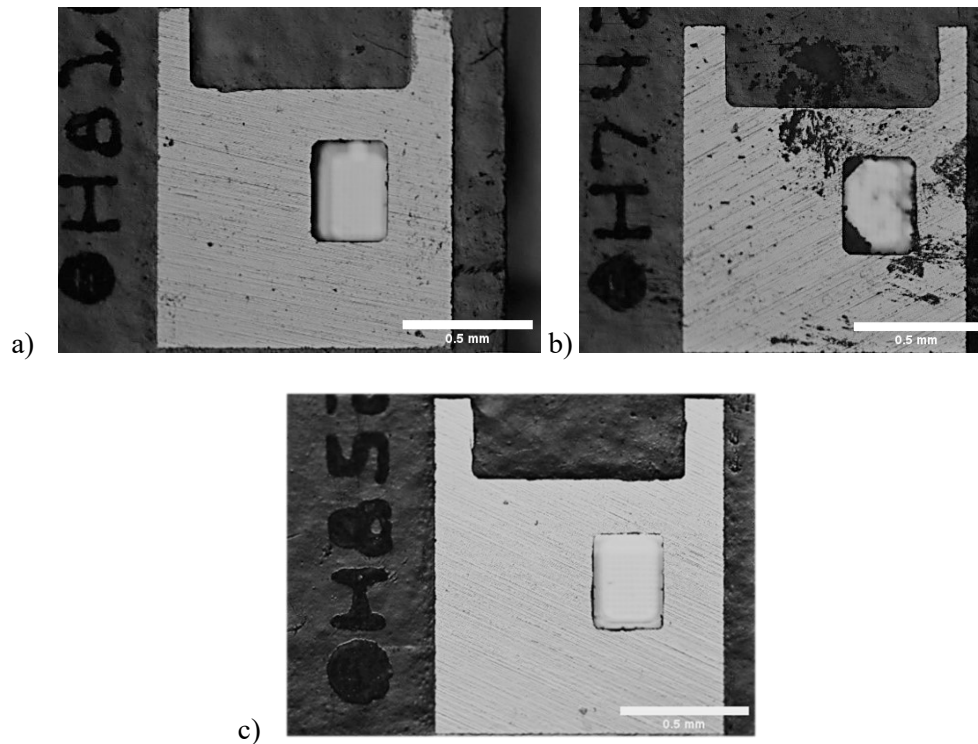


Figure 5-1 Microscopy image of MEMS sensor a) before embedment, b) 28 days in commercially purchased polyethylene terephthalate enclosures and c) 28 days in 3D printed PLA enclosure.

5.2.4 Limitations and improvements

From the preliminary results presented, it was evident that 3D-printed PLA enclosures were capable of preventing particulate contamination during concrete casting and curing. However, it is uncertain if sensor components had degraded at the microscopic scale. To better qualify and quantify the rate of degradation, other advanced analytical techniques can be employed to characterize surface features and examine morphological changes. SEM-BSE analysis can be adopted to evaluate the degradation of silicon thin films, accumulation of unreacted cement and fine aggregates, and cracks on solder joints. SEM-EDS analysis can examine and quantify the electrochemical migration of metal substrates.

5.3 Experimental

5.3.1 Materials and mix proportions

Concrete with a water/cement ratio (w/c) of 0.55 and 0.4, and an aggregate volume fraction of 70 % was prepared. Cement I 52.5 R was used with the chemical and physical properties specified in Section 3.1.1. Mixes were designed with the proportions shown in Table 5-2.

Table 5-2 Mix proportions.

<i>Mix design</i>	<i>w/c ratio</i>	<i>Water (kg/m³)</i>	<i>Sand (kg/m³) <5mm</i>	<i>Coarse aggregate (kg/m³) <10mm</i>	<i>Cement (kg/m³)</i>	<i>Aggregate vol fraction: %</i>
C-0.55-PC-70	0.55	189.1	719.4	1079.1	343.8	70
C-0.4-PC-70	0.4	166.1	719.4	1079.1	415.2	70

5.3.2 Samples

Fourteen specimens with a 100 mm diameter and thickness of 85 mm were prepared with the methods described in Section 4.8. Subsequently, specimens were subjected to drying and wetting by placing in a 50 °C oven for four weeks and incubators containing ammonium nitrate saturated salts at 65% RH for 26 weeks. After 7 months of embedment, RFID MEMS sensors were retrieved by applying pressure to the corners of concrete specimens with a bench vice shown in Figure 5-2.



Figure 5-2 Retrieving RFID MEMS sensors from concrete samples after 7 months of embedment.

5.3.3 Simulated marine environments

To characterize the impact of simulated marine environments, six RFID MEMS sensors were placed on a petri dish above two 5 × 5 mm plastic rods in containers containing shallow water and 3% wt. NaCl salt solution to a depth of 6-7mm (Figure 5-3). Images of each sample were captured at 3 and 7 days, then at 7 day intervals up to 63 days of exposure. Throughout the test, containers were covered with a lid and samples were removed as rapidly as possible to minimise water evaporation and condensation. Care was taken to ensure that the water level was maintained. Two replicates were prepared for each exposure condition.

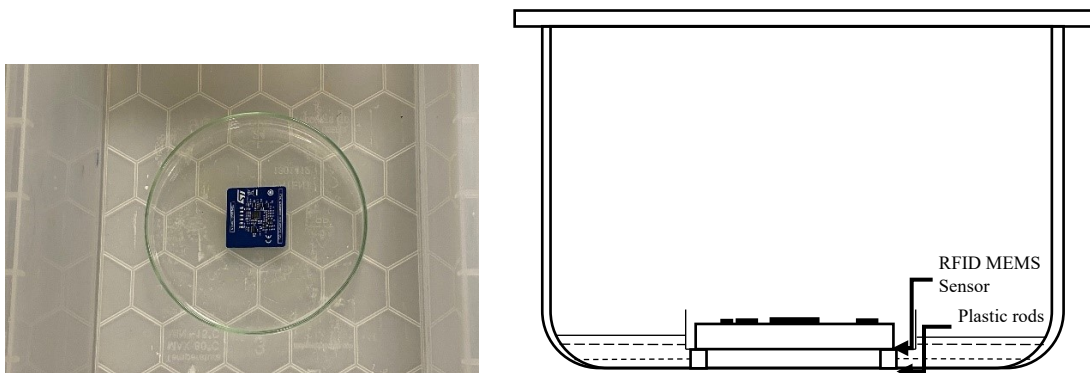


Figure 5-3 Exposing RFID MEMS sensors to 100% RH humidity and 3% wt. NaCl salt solution.

5.3.4 Fatigue of MEMS sensors

Fatigue of silicon thin films was investigated by subjecting ten RFID MEMS sensors to weekly wetting and drying cycles for a year. For wetting cycles, RFID MEMS sensors were exposed to 100% RH environments with a similar setup shown in section 5.3.3. Subsequently, RFID MEMS sensors were dried in a temperature-controlled laboratory at $21 \pm 2^\circ\text{C}$ and $55 \pm 5\%$ RH. The image of each sample was taken after 1 year of exposure and evaluated against a control sample that was not exposed to cyclic wetting and drying. Nine replicates were prepared for fatigue testing.

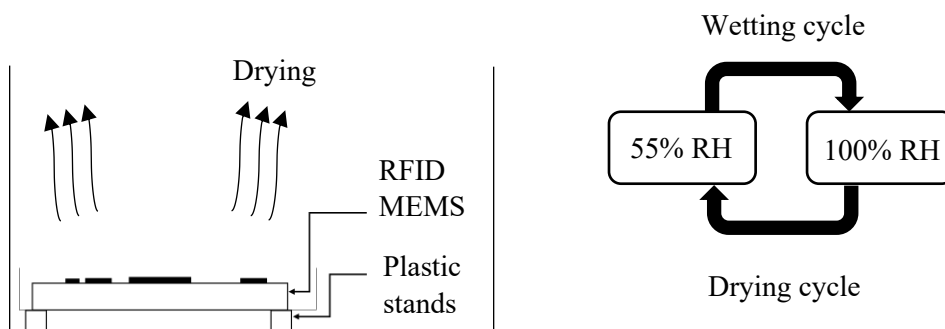


Figure 5-4 Exposing RFID MEMS sensors to wetting and drying cycles to examine fatigue.

5.3.5 Performance test

After exposing RFID MEMS sensors to simulated marine environments, cementitious materials and cycling wetting and drying as described in Sections 5.3.2, 5.3.3 and 5.3.4, the performance of each sensor was tested alongside unexposed control samples. The test consists of placing RFID MEMS sensors in controlled temperature laboratory conditions at $21 \pm 2^\circ\text{C}$ and $55 \pm 5\%$ RH, and enclosure containing moisture (100% RH) at 5 minute intervals with measurements obtained every 5 seconds.

5.3.6 Olympus BX51 stereomicroscopy

The surface morphology of MEMS sensors was observed with an Olympus BX51 stereomicroscope with the equipment and setup shown in Section 3.3.2. Images were captured and further processed with Fiji (ImageJ version 1.53t NIH) by adjusting the colour threshold to red and brightness to 195 (Schindelin et al. 2012; Tan et al. 2016). Each degradation site was segmented and saved as a region of interest (ROI) using the ROI manager tool shown in Figure 5-5 (Eun et al. 2018). To obtain accurate measurements, images were calibrated with known distances and pixel aspect ratio.

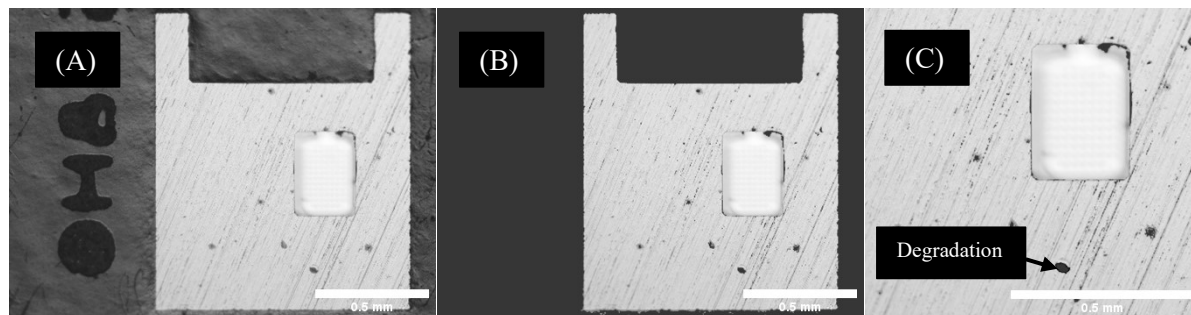


Figure 5-5 a) Adjusting colour threshold of images obtained from stereomicroscope and b) estimating total area of degradation of silicon material.

5.3.7 Backscattered electron microscopy (BSE)/Energy Dispersive Spectroscopy (EDS)

Morphological changes and elemental analysis of sensor components were examined with Hitachi TM4000 Plus scanning electron microscope (SEM) equipped with an Oxford Instruments Aztec

Advanced X-Act Compact energy dispersive X-ray detector (EDS) shown in Section 3.2. The SEM was operated at 15 kV in backscattered electron (BSE) mode with a working distance of 7 mm to achieve an optimal spatial resolution (Yio, Wong, and Buenfeld 2016). A minimum of 20 spot EDS analysis was conducted for each silicon degradation site with a spacing of 0.06 mm and the maximum carbon counts presented (Figure 5-8). The maximum area of degradation was measured by adjusting the colour threshold to red and brightness to 160. Each degraded area was segmented and saved as a region of interest (ROI) using the ROI manager tool shown in Figure 5-6 (Eun et al. 2018). Additionally, coatings and solder joints of resistors and capacitors were studied with energy dispersive spectroscopy (EDS) indicated with dotted lines shown in Figure 5-7. Areas of voids were measured by a tracing tool with a total of 15 measurements taken and the overall average value presented.

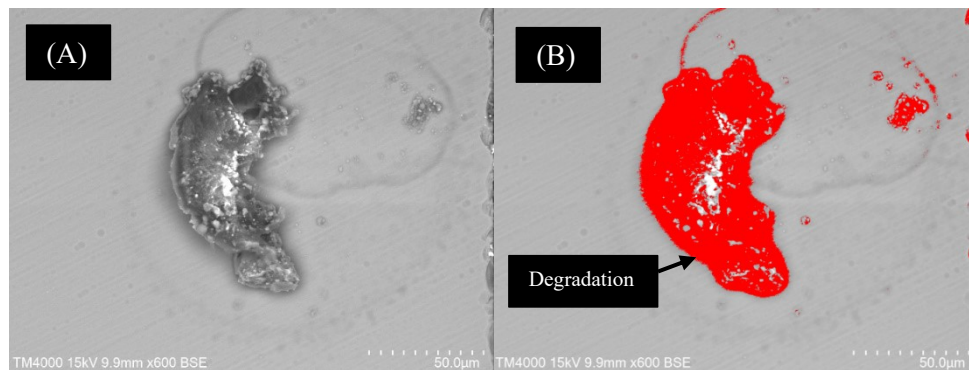


Figure 5-6 Adjusting colour threshold of images a) obtained from backscattered electron microscopy and b) estimated area of degraded silicon material.

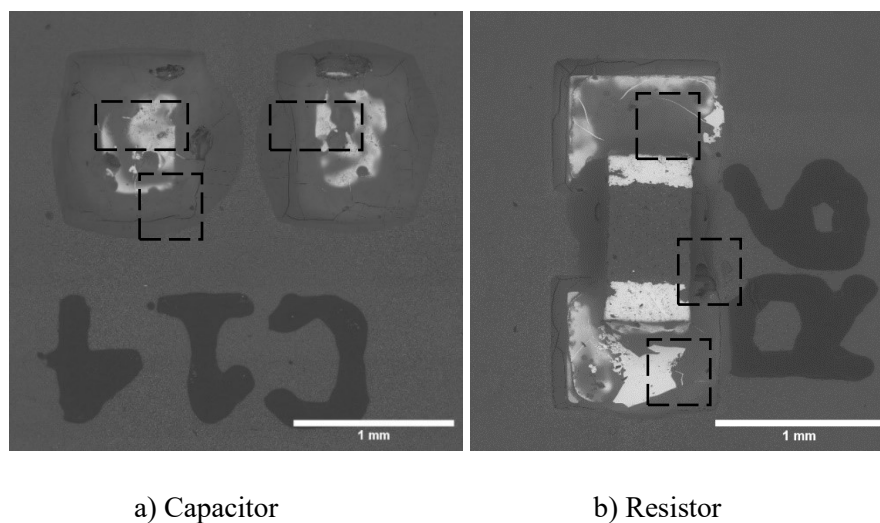


Figure 5-7 Examining coatings and solder joints of a) capacitors and b) resistors.

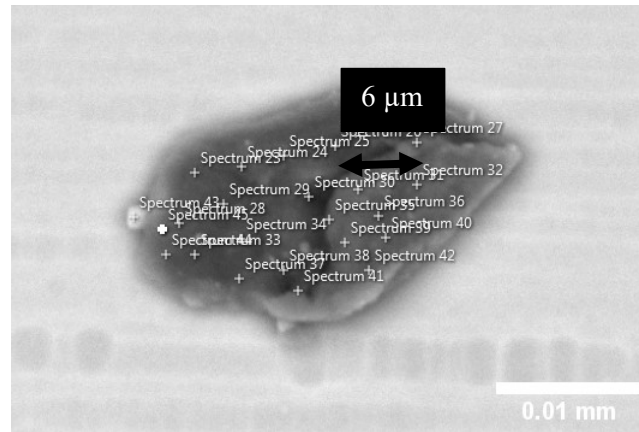


Figure 5-8 Analysing degradation site on silicon thin films.

5.4 Results

5.4.1 Surface morphology vs. performance

5.4.1.1 Influence of cementitious materials

Figures 5-9 and 5-10, present surface morphology images and relative humidity measurements before and after embedding in low and high-strength concrete. To produce these figures, samples of identical curing age and water to cement ratio were grouped. The Y-axes and X-axes were plotted on similar scales to enhance clarity.

From the optical images captured, no signs of particulate contamination were observed on MEMS sensors after 7 months of embedment. These findings were in agreement with the results presented in Section 5.2.3. Nonetheless, silicon thin films have significantly degraded for 3 and 14 days cured low-strength concrete specimens as seen in Figure 5-9 a and Figure 5-9 c. This is expected because samples with a higher water to cement ratio have a medium of lower viscosity; thus, allowing cement particles and fine aggregates to accumulate on silicon thin films. Moreover, similar relative humidity measurements were obtained for both exposed and unexposed sensors. This indicates that the overall performance was unaffected and RFID MEMS sensors were functional after 7 months of embedment in concrete. However, there is a slight discrepancy shown in Figure 5-10 d. This can be caused by slight changes in laboratory temperatures while obtaining measurements.

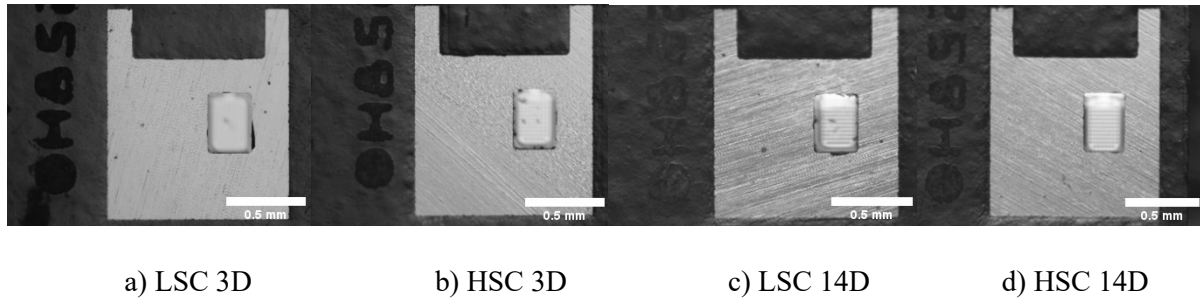


Figure 5-9 Examining the surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 after embedding in low and high strength concrete for 7 months subjected to wetting and drying cycles.

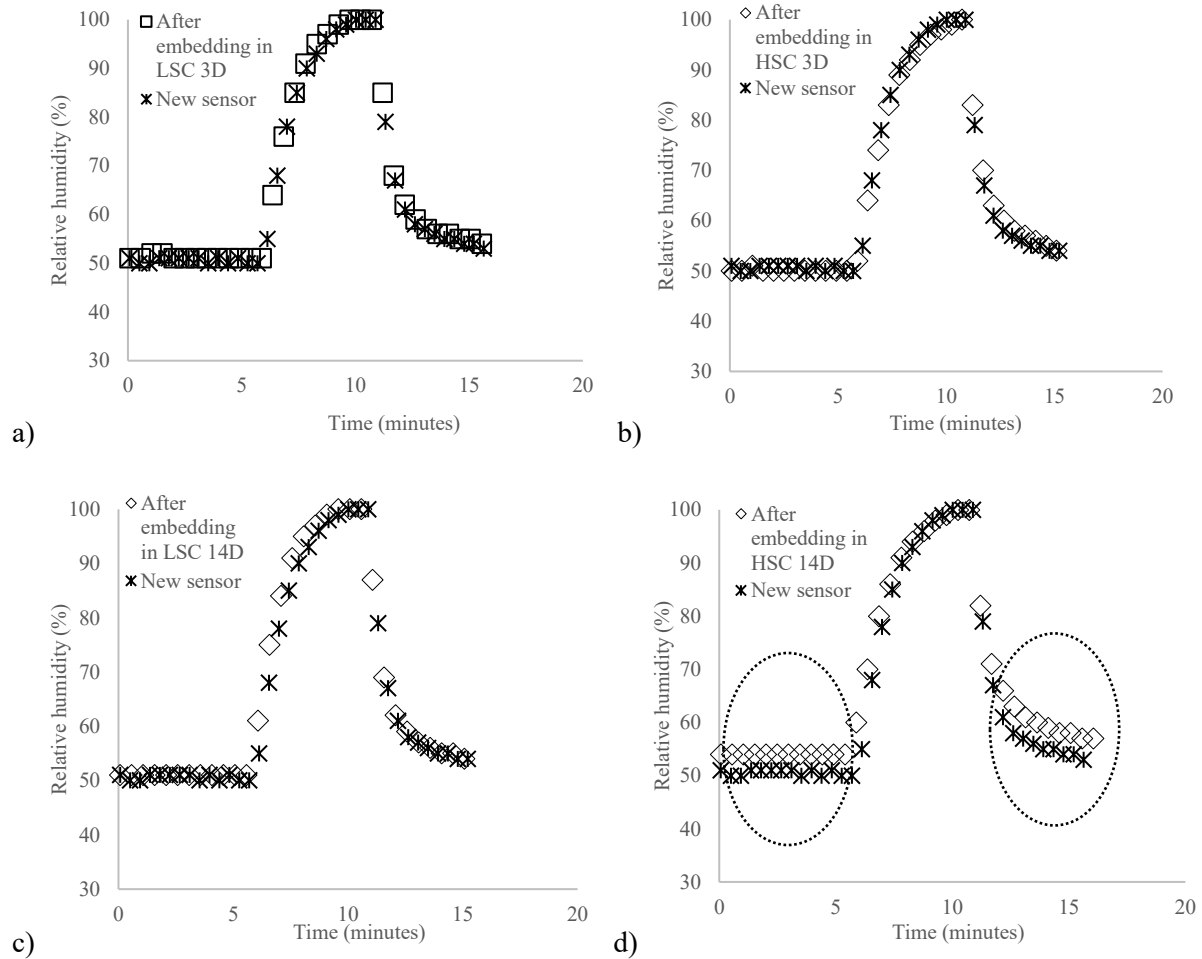


Figure 5-10 Performance of RFID MEMS sensors after embedding in low and high strength concrete for 7 months subjected to wetting and drying cycles.

5.4.1.2 Direct exposure to 100% RH and 3% wt. NaCl

Figures 5-11 to 5-14, present surface morphology images and relative humidity measurements before and after direct exposure to 100% RH and 3% wt. NaCl salt solution. To develop a clear analysis, results with identical exposure conditions were sorted into groups. Additionally, Y-axes and X-axes were plotted on similar scales to enhance clarity.

From the results obtained, it was observed that silicon thin films had degraded after 4 days of exposure for both 100% RH and 3% wt. NaCl salt solution (Figures 5-11 and 5-13). This was expected because corrosive elements from electronic components can accumulate on silicon thin films with the presence of moisture (Noh et al. 2009; Noh, Lee, and Jung 2008). In addition, the presence of chloride ions can cause silicon to corrode and oxidize at a faster rate. On the other hand, it was interesting to observe silicon thin films degrading at a slower rate after 49 days of exposure. This suggests that the rate of degradation has approached a state of equilibrium.

Nevertheless, identical relative humidity measurements were obtained for RFID MEMS sensors exposed to 100% RH for 63 days. This suggests that reproducible measurements can be attained after subjecting RFID MEMS sensors to moisture for long periods of time. However, a difference in the rate of response was observed at 14 and 63 days of exposure (Figure 5-12 b and d). By exposing MEMS sensors to 3% wt. NaCl salt solution for 63 days, a difference of 11% in relative humidity was measured. This can suggest an increase in sensitivity of MEMS sensors.

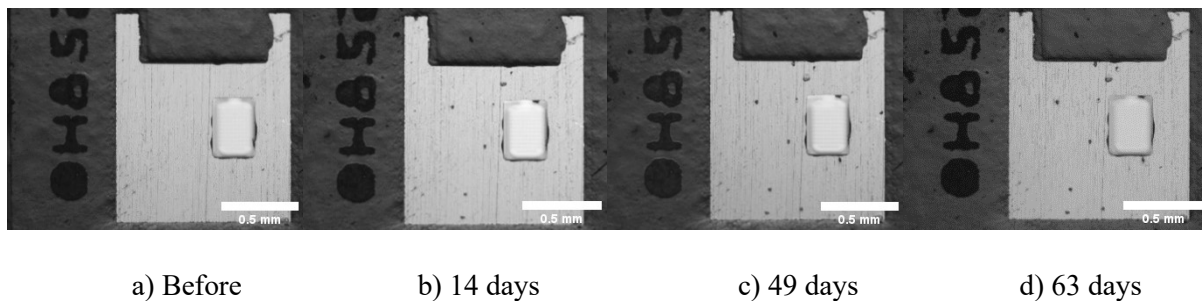


Figure 5-11 Examining the surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 after direct exposure to 100 % RH for 63 days.

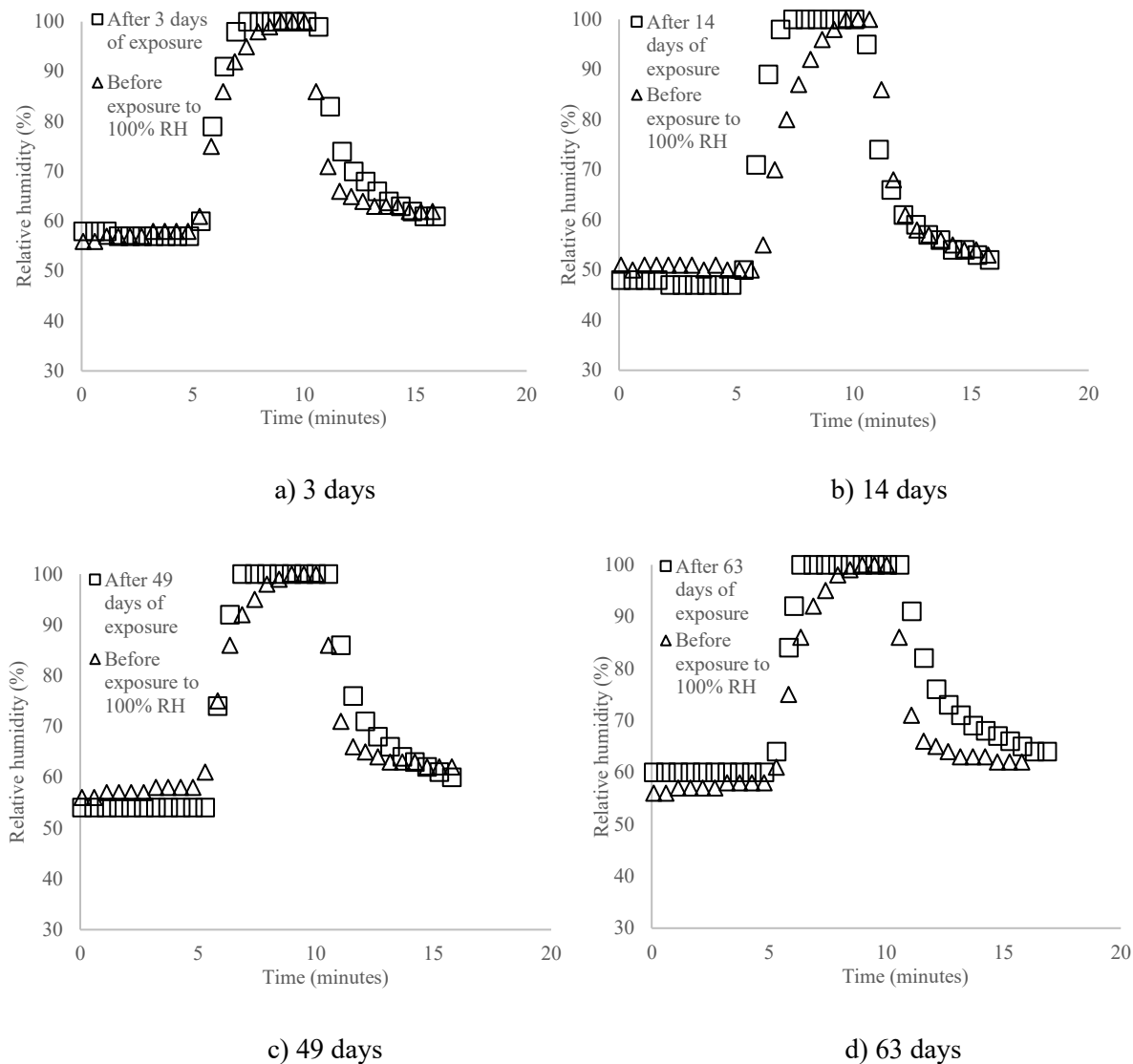


Figure 5-12 Performance of RFID MEMS sensors after direct exposure to 100% RH for 63 days.

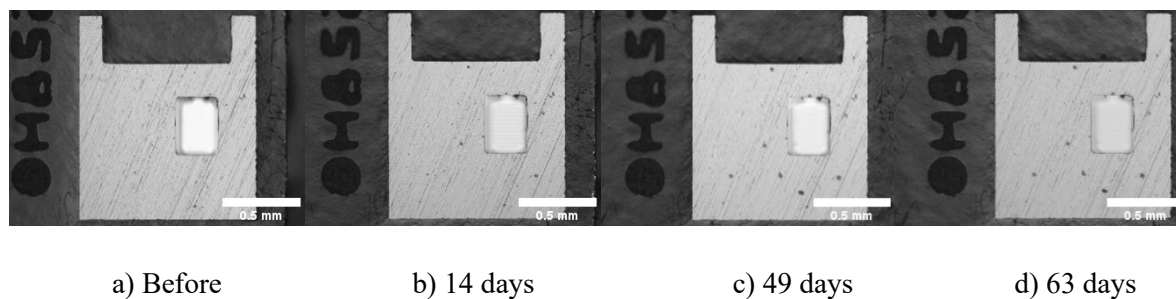


Figure 5-13 Examining the surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 after direct exposure to 3% wt. NaCl salt solution for 63 days.

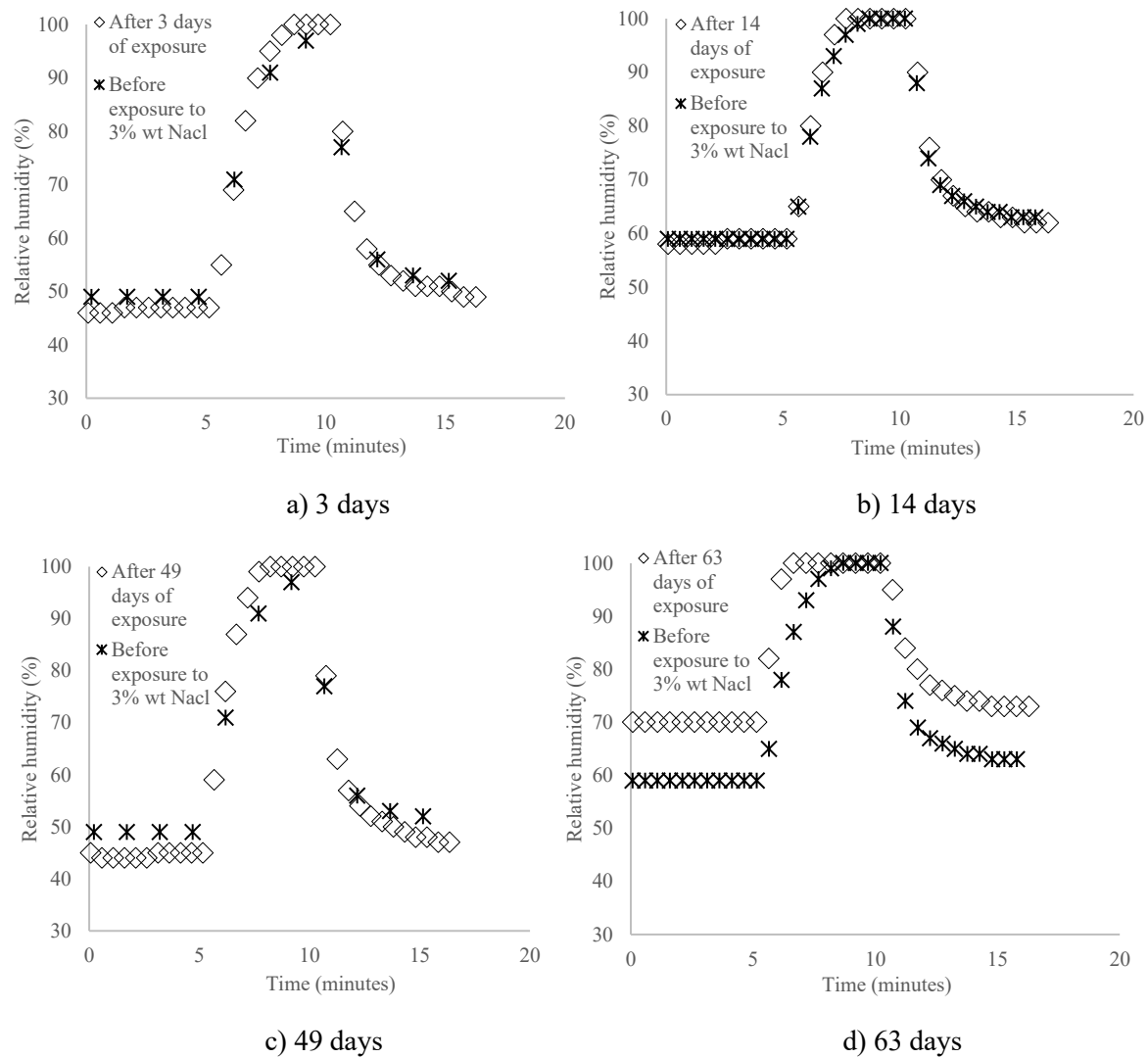
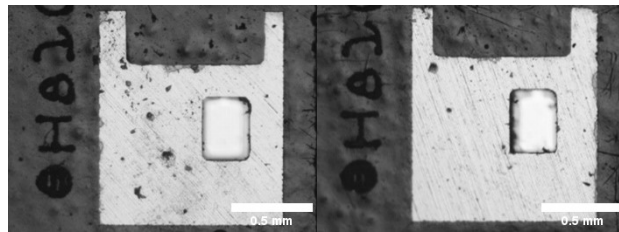


Figure 5-14 Performance of RFID MEMS sensors after direct exposure to 3% wt. NaCl salt solution for 63 days.

5.4.1.3 Fatigue of MEMS sensors

Figures 5-15 and 5-16, present surface morphology images and relative humidity measurements before and after one year of cyclic wetting and drying. For better quantification, Y-axes and X-axes were plotted on similar scales.

From the images captured, it was evident that silicon thin films have degraded after 1 year of exposure to cyclic wetting and drying cycles. This was similar to findings presented in Section 5.4.1.2, where degradation was observed after 4 days of exposure to moisture. Moreover, cracks and voids were discovered on the surfaces of silicon carbide microchips. This indicates the deterioration and fatigue of silicon carbide material when exposed to moisture for long periods of time. Nonetheless, identical relative humidity measurements were obtained (Figure 5-16). This implies that the overall performance was unaffected and reproducible measurements can be attained after subjecting RFID MEMS sensors to cyclic wetting and drying.



a) Sensor 1

b) Sensor 2

Figure 5-15 Examining surface morphology of MEMS sensors under a stereomicroscope Olympus BX51 subjected to 1 year of cyclic wetting and drying.

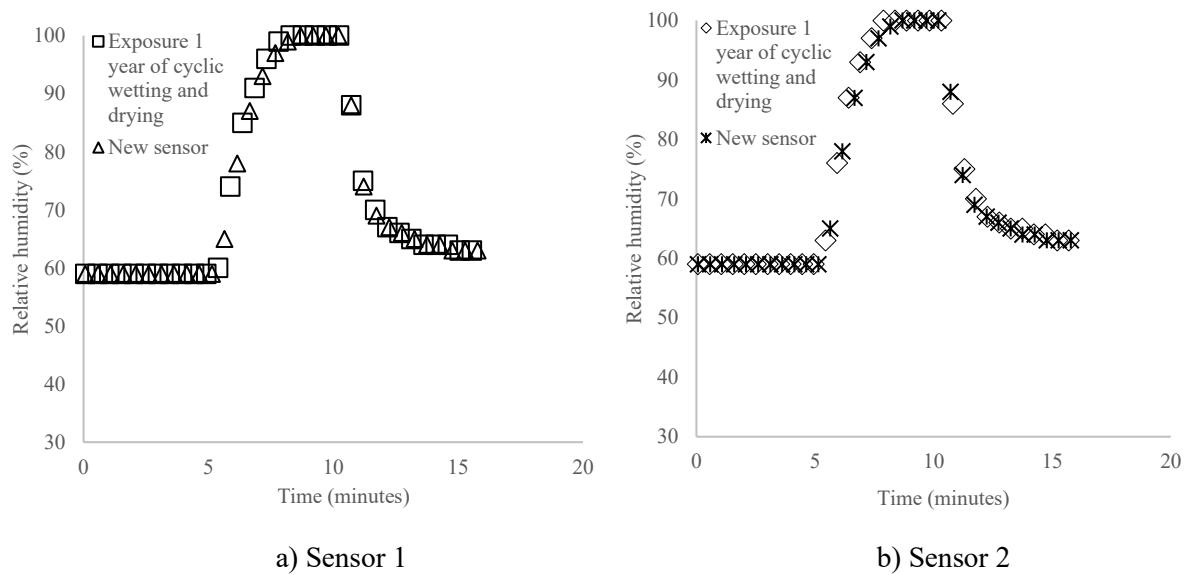


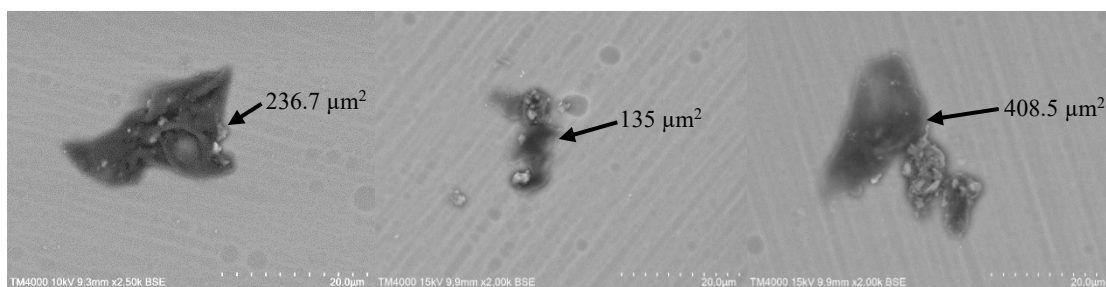
Figure 5-16 Performance of MEMS sensors after being subjected to 1 year of cyclic wetting and drying.

5.4.2 Particulate and contamination analysis

Figures 5-17 and 5-18, present SEM images and elemental analysis of RFID MEMS sensors after embedding in concrete, and exposure to simulated marine environments. Results obtained from identical analytical techniques were sorted into groups with Y-axes and X-axes plotted on similar scales to enhance clarity.

By examining the surface characteristics of silicon thin films, results indicate that low-strength concrete specimens have a greater area of degradation compared to high-strength concrete. An area of $408.5 \mu\text{m}^2$ was measured for low-strength concrete samples cured for 14 days. In comparison, an area of $197.2 \mu\text{m}^2$ was measured for high-strength concrete samples cured for 14 days. This is in agreement with optical images presented in Section 5.4.1.1, where signs of degradation were observed on silicon thin films for low-strength concrete specimens. Moreover, direct exposure to 3% wt. NaCl salt solutions had the greatest area of degradation of $4000 \mu\text{m}^2$.

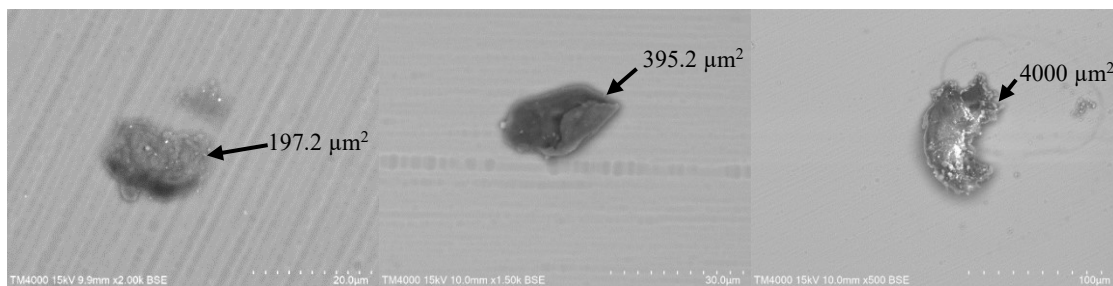
Results obtained from EDS analysis indicated potassium, sodium and calcium elements on surfaces of silicon thin films for low-strength concrete samples. This suggests the degradation of silicon materials close to cement paste pore solution. In addition, higher counts of carbon were measured after exposing silicon thin films to 100% RH and 3% wt. NaCl which suggests the deterioration of silicon thin films and exposure of silicon carbide material.



a) LSC 3D- 7 months

b) HSC 3D- 7 months

c) LSC 14D- 7 months

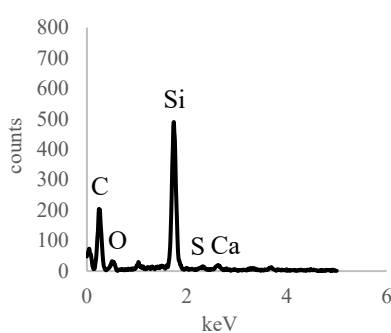


d) HSC 14D- 7 months

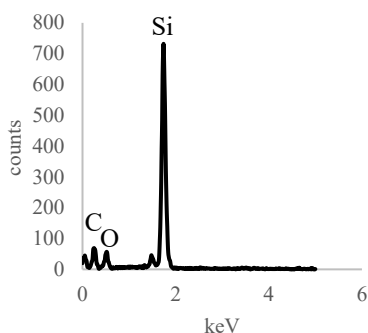
e) 100% RH- 2 months

f) 3% wt. NaCl- 2 months

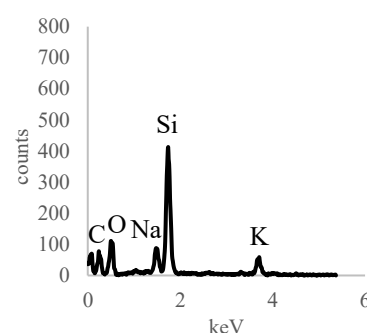
Figure 5-17 Examining degradation of silicon thin film exposed to cementitious materials and simulated environments with SEM-BSE.



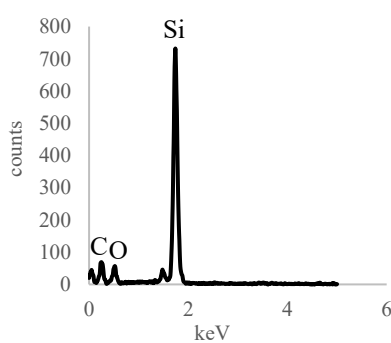
a) LSC 3D- 7 months



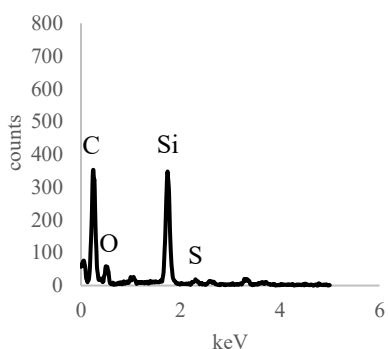
b) HSC 3D- 7 months



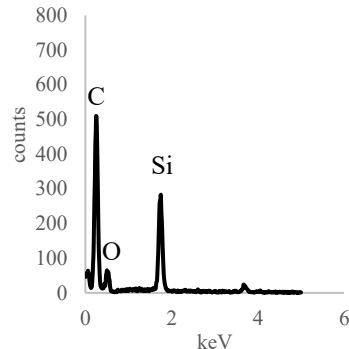
c) LSC 14D- 7 months



d) HSC 14D- 7 months



e) 100% RH- 2 months



f) 3% wt. NaCl- 2 months

Figure 5-18 Elemental analysis of degraded material after exposure to cementitious materials and simulated environments with SEM-EDS.

5.4.3 Area of degradation

Figure 5-19, presents the total area of degradation against elapsed time for RFID MEMS sensors exposed to 100% RH and 3% wt. NaCl salt solution for 2 months. Measurements with identical exposure times were sorted into groups to enhance clarity.

The overall trend suggests that silicon thin films subjected to moisture degraded at a faster rate during the initial exposure period. From 3 to 49 days of exposure, a total degradation area of 6465 μm^2 was measured for 100% RH compared to 5704 μm^2 for 3% wt. NaCl salt solution. However, the rate of degradation equilibrated and stabilized after 49 days with an increase of 207.54 μm^2 at 56 days. This was similar to findings presented in Section 5.4.1.2, where changes to surface characteristics were significantly reduced. On the contrary, an increase of 2103.81 μm^2 was measured after 56 days of exposure to 3% wt. NaCl salt solution.

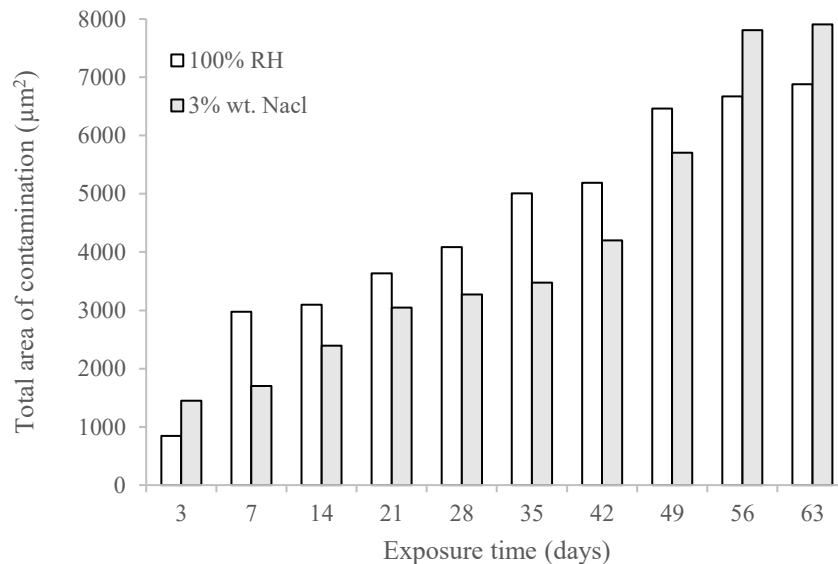


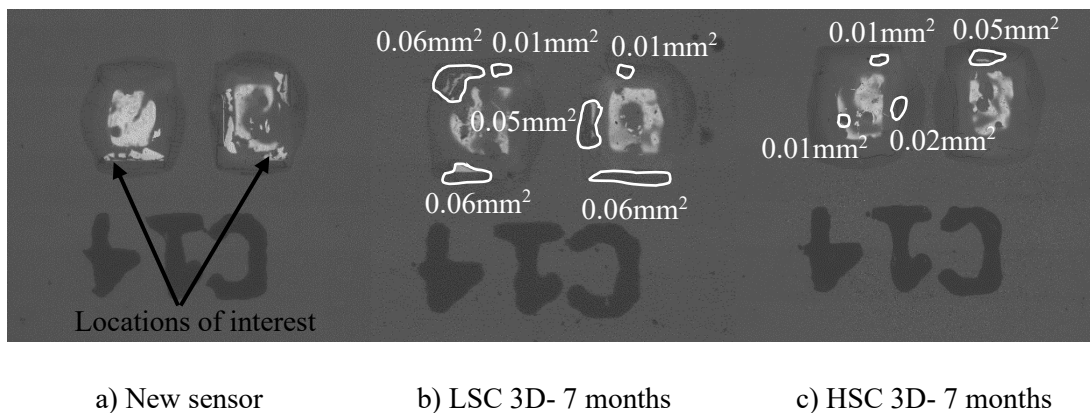
Figure 5-19 Estimating the total area of degradation of silicon thin films over time after direct exposure to 3% wt. NaCl salt solution and 100% RH for 2 months.

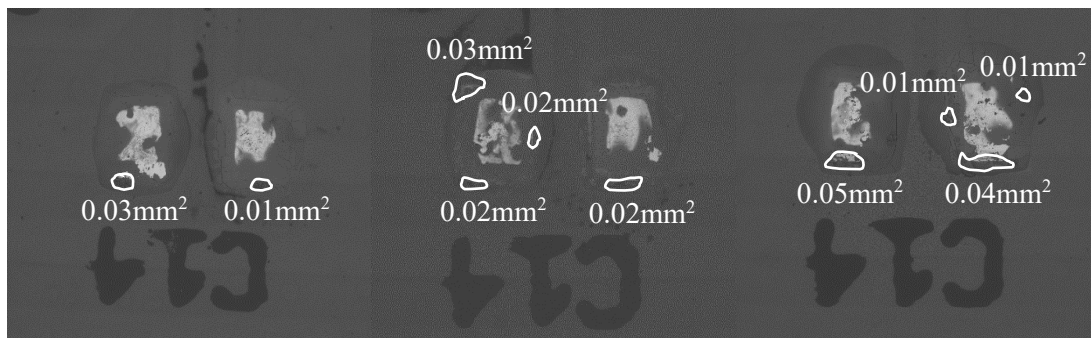
5.4.4 Ageing of electronic components

5.4.4.1 Capacitor

Figures 5-20 and 5-21, present SEM images and elemental analysis of capacitors after embedding in concrete, exposure to simulated marine environments and cyclic wetting and drying. To enhance clarity, measurements obtained from identical analytical techniques were sorted into groups with Y-axes and X-axes plotted on similar scales.

Results indicate capacitors age at a faster rate after embedding in concrete with voids formed on surfaces and exposure of internal electronic components shown in Figure 5-21. In comparison, no signs of degradation were observed for specimens directly exposed to 100% RH and 3% wt. NaCl salt solution. This was expected because sensor components were subjected to environments of high alkalinity. In addition, a reduction in carbon counts was measured for specimens embedded in low-strength concrete compared to high-strength concrete. Carbon counts of 105 and 197 were measured for low-strength concrete specimens cured for 3 and 14 days. On the other hand, carbon counts of 282 and 306 were measured for high-strength concrete specimens cured for 3 and 14 days. This might suggest the deterioration of capacitors from the exposure to cement paste pore solution and is in agreement with optical images and particulate analysis results presented in Sections 5.4.1.1 and 5.4.2.

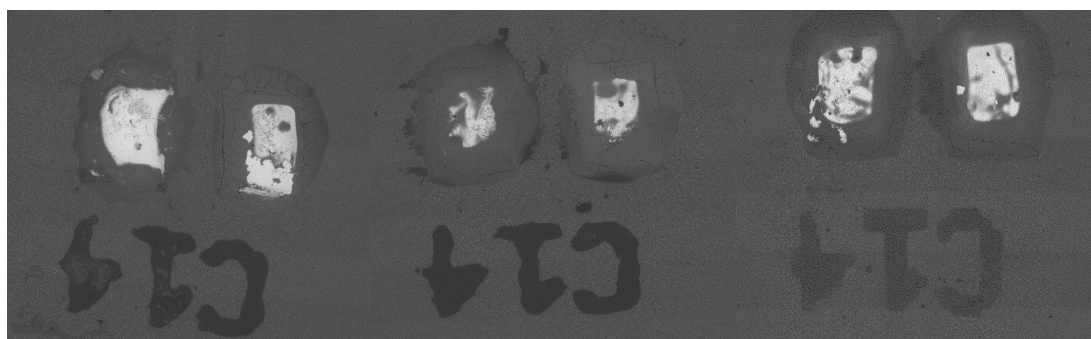




d) LSC 14D- 7 months

e) HSC 14D- 7 months

f) Improper compaction

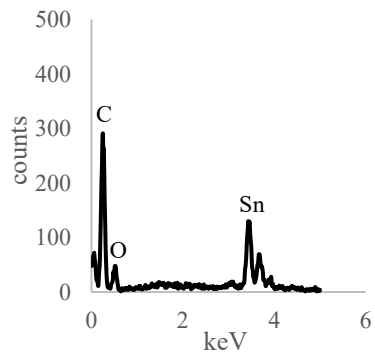


g) 100% RH- 2 months

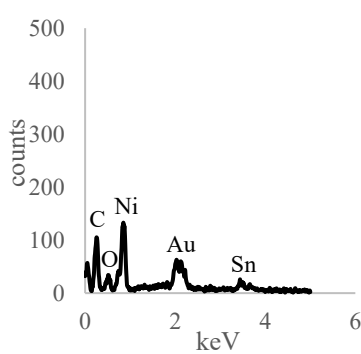
h) 3% wt. NaCl- 2 months

i) Wetting and drying- 1 year

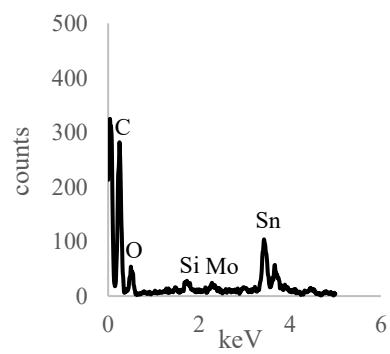
Figure 5-20 Examining capacitor after exposure to cementitious materials and simulated environments with SEM BSE.



a) New sensor



b) LSC 3D- 7 months



c) HSC 3D- 7 months

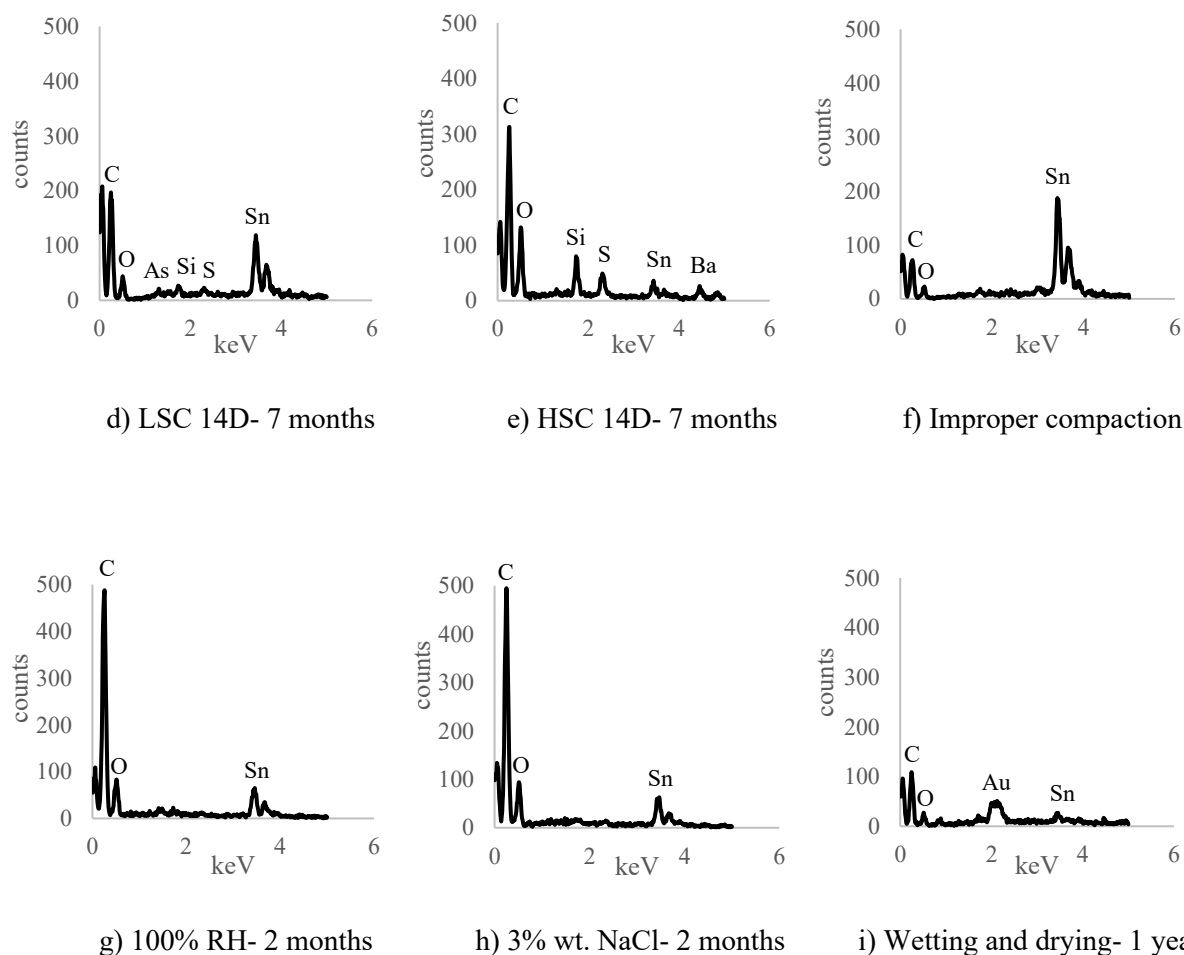


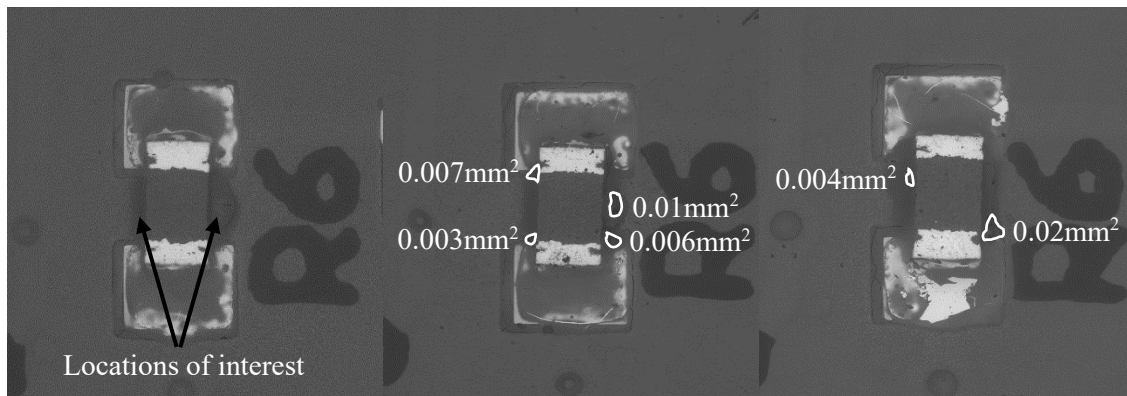
Figure 5-21 Elemental analysis of capacitor after exposure to cementitious materials and simulated environments with SEM-EDS.

5.4.4.2 Resistor

Figures 5-22 and 5-23, present SEM images and elemental analysis of resistors after embedding in concrete, exposure to simulated marine environments and cyclic wetting and drying. Figures with identical analytical techniques were sorted into groups with Y-axes and X-axes plotted on similar scales for better quantification.

From the images presented, a greater quantity of voids was found alongside resistors and between solder joints after embedding in concrete for 7 months. The total area of voids measured for low-strength concrete cured for 3 and 14 days was 0.026 mm² and 0.04 mm². Similarly, the total area of voids measured for high-strength concrete cured for 3 and 14 days was 0.02 mm² and 0.06 mm². These findings

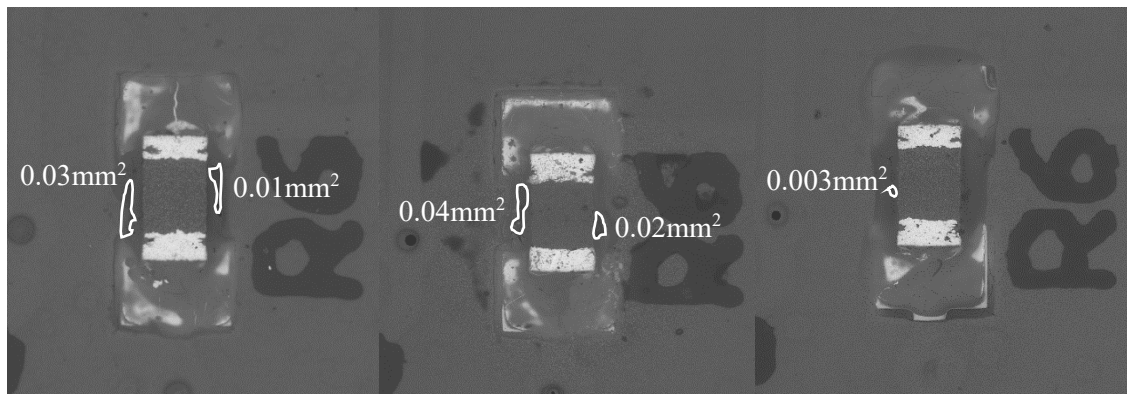
are in agreement with the results presented in Section 5.4.4.1 indicating sensor components degrade at faster rates after embedment in concrete. Furthermore, an increase in carbon counts was observed on solder joints, suggesting the transport and degradation of silicon carbide materials. Additionally, barium and sulphur elements were identified for concrete specimens with insufficient compaction indicating the degradation of printed circuit boards (PCB).



a) New sensor

b) LSC 3D- 7 months

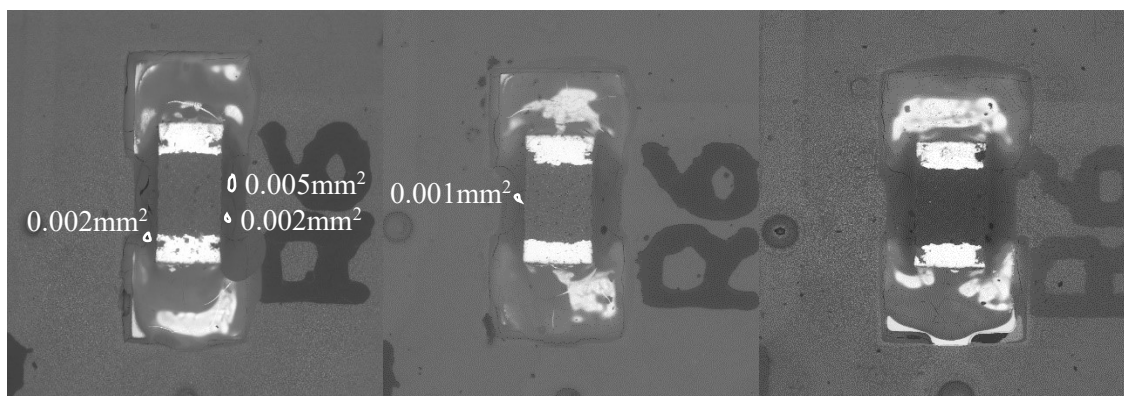
c) HSC 3D- 7 months



d) LSC 14D- 7 months

e) HSC 14D- 7 months

f) Improper compaction

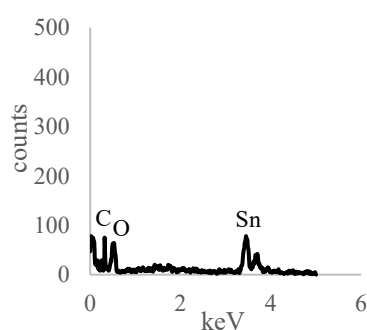


g) 100% RH- 2 months

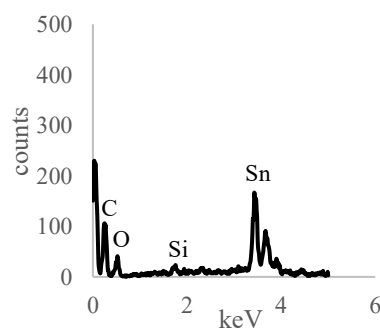
h) 3% wt. NaCl- 2 months

i) Wetting and drying- 1 year

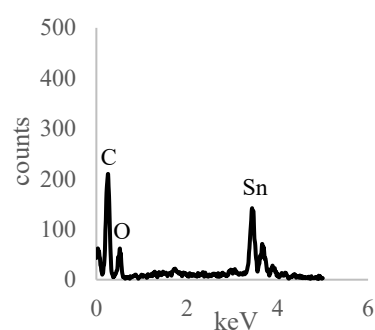
Figure 5-22 Examining soldering joints of resistor after exposure to cementitious materials and simulated environments with SEM BSE.



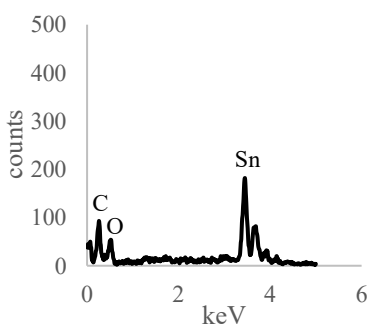
a) New sensor



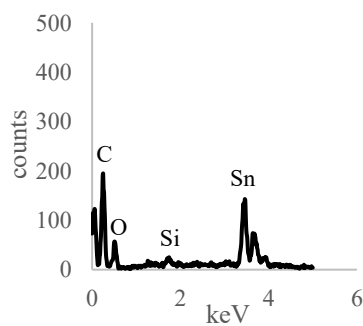
b) LSC 3D- 7 months



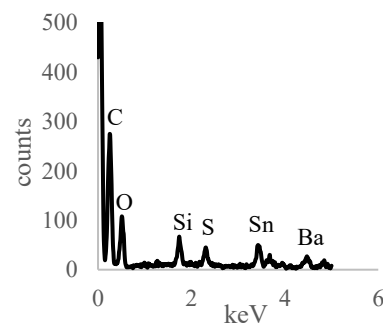
c) HSC 3D- 7 months



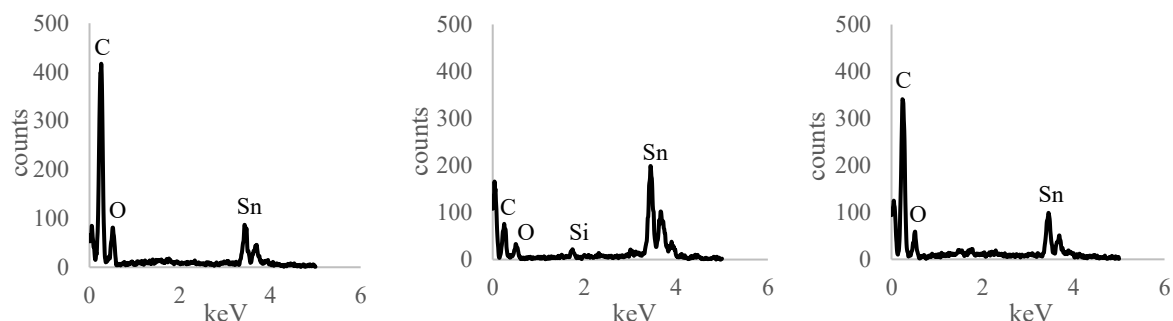
d) LSC 14D- 7 months



e) HSC 14D- 7 months



f) Improper compaction



g) 100% RH- 2 months

h) 3% wt. NaCl- 2 months

i) Wetting and drying- 1 year

Figure 5-23 Elemental analysis of solder joints after exposure to cementitious materials and simulated environments with SEM-EDS.

5.5 Discussion

5.5.1 Durability and performance of MEMS sensors

It is critical to inspect and identify durability and performance issues of RFID MEMS sensors during the development of monitoring techniques. These issues can originate from fabrication processes where spin-coating and photolithography will produce polymer structures with non-uniform thickness ranging from 15.62 – 19.35 μm as a result of inconsistent spin speeds (Y. Li 2018; Y. Li et al. 2013). Plasma exposure can cause a layer of hard crust (Novolac-Diazoquinone-based positive photoresists) to form and cause failures (Joubert et al. 1991). These imperfections were identified from images presented in Figure 5-1 a, but relative humidity measurements obtained from this study suggest that the overall performance was unaffected.

Additionally, corrosive and moist environments can degrade silicon thin films and cause RFID MEMS sensors to underperform. For this study, RFID MEMS sensors were directly exposed to 3% wt. NaCl salt solutions and 100% RH for 2 months. At 14 days of exposure, significant degradation was observed on

silicon thin films with identical relative humidity measurements obtained. By increasing the exposure time to 63 days, a difference in relative humidity measurements was obtained for specimens exposed to 3% wt. NaCl salt solutions. In contrast, performance was unaffected by exposing MEMS sensors to 100 % RH environments. These findings are similar to studies conducted by Broas et al. (2015) with results indicating a reduction in fracture strength for silicon nitride film exposed to Cl₂, H₂S, NO₂ and SO₂ gases for 50 days compared to 95% RH at 85 °C for 140 days. Results from elemental analysis have shown an increase in carbon counts indicating the exposure of silicon carbide material shown in Figure 5-24.

Furthermore, environments of high alkalinity, pH 12.5 - 13, can damage sensor components and influence the performance of sensors (Deschner et al. 2012; Strangfeld, Johann, and Bartholmai 2019). The presence of OH⁻ ions can dissolve silicon thin films and form Si-OH complexes (Starosvetsky, Kovler, and Ein-Eli 2004). Optical images and relative humidity measurements obtained from this study suggest silicon thin films and sensor components deteriorate at faster rates after exposure to cementitious materials without having a significant impact on performance.

5.5.2 The effects of particulate contamination on silicon thin films

Other complications might arise when embedding RFID MEMS sensors in concrete. Particulates can be transported and deposited on silicon thin films producing unreliable measurements. In particular, cement particles have a length scale of 20-60 µm (Erdogan et al. 2010). Thus, it is probable that cement particles can contaminate surfaces and cause micro-machines to malfunction (D. M. Tanner et al. 2000). For this study, degradation was observed at locations close to cementitious material with potassium, sodium and calcium elements found on surfaces of silicon thin films. These findings are in agreement with studies conducted by Starosvetsky et al. (2004) with Atomic force microscopy (AFM) images indicating the dissolution of silicon and reduction of silicon surface atoms after the exposure to 12 - 28 % wt. NaOH solutions at 25 °C and 60 °C.

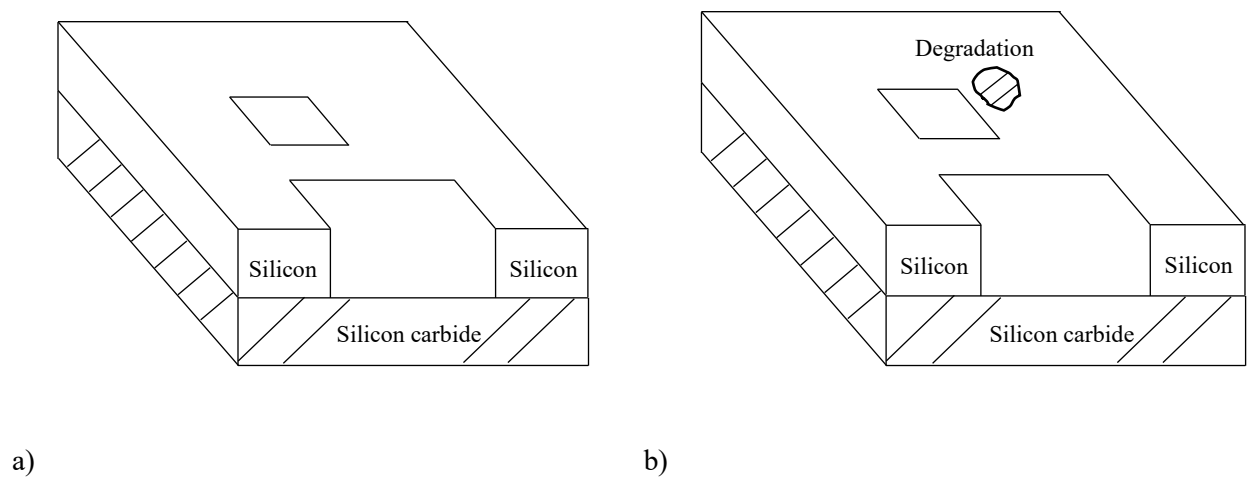


Figure 5-24 Figure to illustrate materials and structure of RFID MEMS sensor a) before and b) after degradation.

5.5.3 Durability of electronic components

During in-situ monitoring, sensor components provide the functionality and usability for wireless communication and monitoring. However, flexural cracks can develop on ceramic capacitors over time allowing moisture to weaken bonds between ceramics and polymers, and cause failures (Alam, Azarian, and Pecht 2012). Findings from this study indicate capacitors age at a faster rate after embedding in concrete with voids formed on surfaces and exposure of internal electronic components. Furthermore, gold elements were discovered on surfaces after one year of cyclic wetting and drying. This might indicate the migration of metals after long-term exposure to moisture. Similar findings were presented by experimental studies conducted by Noh et al. (2008). Electrochemical migration of dendrites consisting of gold and small amounts of nickel and copper was found across gold and nickel electrodes after applying a voltage of 3 to 18 V with a droplet of distilled water placed on printed circuit boards.

On other hand, the formation of intermetallic compounds and voids at solder joints can result in embrittlement and reduction in fatigue life. Results from this study have indicated the formation of voids and cracks on resistors and solder joints after the embedment in concrete. This implies that environments with high alkalinity can oxidise and dissolve solder material underneath resistors with an overall increase in surface voids (Seo et al. 2019). These findings were similar to experimental studies conducted by

Muhammad et al. (2015). They observed the formation of new oxide phases of SnO and SnO₂ after exposing Sn-9Zn solder specimens to a 6 M KOH electrolyte.

5.6 Conclusions

The reliability and performance of RFID MEMS sensors were investigated. This includes long-term embedment in low and high-strength concrete specimens for 7 months, 2 months of direct exposure to 3% wt. NaCl salt solution and 100% relative humidity, and 1 year of cyclic wetting and drying to examine fatigue. The study was an attempt to explore the effects of cementitious material, moisture and corrosive environments on RFID MEMS sensors, with the overall aim of better understanding the impact on reliability and performance.

The main findings are as follows:

- a) Performance of RFID MEMS sensors was not affected after embedding in low and high-strength concrete samples for 7 months. However, a difference in relative humidity measurements was observed after direct exposure to 3% wt. NaCl salt solutions for 63 days.
- b) Silicon thin films had deteriorated at faster rates for low-strength concrete compared to high-strength concrete and degradation was observed at locations close to potassium, sodium and calcium elements found on surfaces of silicon thin films.
- c) After embedding specimens in concrete, voids were found on capacitors and resistors. This can be attributed to the exposure to environments with high alkalinity. Moreover, elemental analysis revealed the exposure of circuitry components with the presence of silicon, gold, nickel and sulfur elements.
- d) Degradation was observed on MEMS sensors after 1 year of cycling wetting and drying. However, performance was unaffected with similar readings when compared to an unexposed sensor.

Chapter 6 Characterising moisture state of air voids in cement-based materials subjected to wetting-drying using RFID-MEMS sensors

This chapter reports on the feasibility and capability of RFID MEMS sensors to monitor changes in the relative humidity of simulated air voids. Considerations of techniques used to develop air voids including limitations, influence on transport properties and application will also be discussed. New protocols were created to study the influence of geometrical size and surface cracks. The tests involved conditioning 25 mm thick cement paste specimens at 50 °C oven, 55% and 75% relative humidity environments to produce varying degrees of microcracks and moisture content. Relative humidity, cumulative absorption and desorption, and microcracks were measured at different water to cement ratios and curing ages to better understand the moisture state of macroscopic voids exposed to wetting and drying.

6.1 Introduction

The steel-concrete interface may contain macroscopic voids such as entrapped air, settlement, bleeding zones and mechanical damage such as cracks, slip and separation. These macroscopic voids are known to influence the integrity and durability of reinforced concrete structures. For example, the reduction of bond strength. In particular, the moisture state of macroscopic voids can critically influence corrosion initiation and substantially affect corrosion rates. Therefore, the capability to monitor the relative humidity of macroscopic voids is highly beneficial for many research and field applications.

A review of literature has shown an inadequate understanding of the relationships between the moisture state of macroscopic voids and corrosion initiation with contradictory findings and opinions (2.2). Macroscopic air voids are often entrapped and surrounded by cement paste, thus; it is plausible that water will preferentially saturate finer pores with higher capillary pressure resulting in longer saturation times. This was modelled by Fagerlund (1993) based on the assumption that capillary pores are completely saturated and capillary pressure in finer pores remains high. Recently, it was incorporated into a model developed by Smith et al. (2020) to examine the influence of cementitious materials on air dissolution times in spherical voids. A spherical air void with a radius of 100µm was predicted to achieve full

dissolution after 100 days. However, it provides no insight into how moisture is being transported into air voids and the influence of vapour diffusion on moisture conditions is unknown (6.2). This is resulted by the lack of non-destructive techniques for characterising the moisture state in air voids, which supports the need for this work (U. M. Angst et al. 2017; Hong S. Wong et al. 2022). The technical challenge is to provide a direct measurement of relative humidity in macroscopic voids with diameters ranging from 1-15mm. Appropriate sensor technologies with reasonable accuracy and high sensitivity are needed to detect changes in moisture content.

This study aims to enhance the understanding of the moisture state of macroscopic voids subjected to wetting and drying. Air voids of various dimensions were created in cement paste specimens which were then dried to equilibrium. A simple experimental method was developed using RFID MEMS sensors to monitor relative humidity. Surface morphology was examined and related to water absorption characteristics to explain the internal relative humidity measurements.

6.2 Theoretical background

According to Fagerlund (1993), water will preferably penetrate through finer pores with a high capillary pressure rather than coarse pores with a lower capillary pressure during capillary suction. Hence, coarse pores will be filled with air as long as the system does not equilibrate. Due to over-pressure, entrapped air in voids with a radius greater than 10 μm will contract and a thin layer of water film will form on the surface. However, this is based on the assumption that capillary pores are completely saturated with water and capillary pressure in finer pores remains high. Under some circumstances, vapour diffusion can transport moisture at a faster rate than capillary absorption and potentially influence the moisture state of voids. During capillary absorption, a greater volume of air will be entrapped in larger voids compared to smaller voids and result in a slower rate of dissolution. By considering Laplace and Boyle's Law, the amount of compressed air in a spherical void with dimensions based on reported laboratory and site data can be computed based on Equation 6-1-5 (1993). Shown in Figure 6-1, voids with a radius of 100 μm will consist of 0.64 % compressed air and 99.36 % filled with water vapour (saturated air) compared to voids with a radius of 10mm with 76.1 % compressed air and 23.9 % filled with water vapour.

By assuming the conservation of mass between atmospheric air and compressed air

$$\frac{4}{3}\pi R^3 P = \frac{4}{3}\pi r^3 P^* \quad 6-1$$

R : radii of spherical air void (m); r : radii of compressed air (m); P : atmospheric pressure (10^5 pa);

P^* : pressure of compressed air

$$P^* = P + \Delta P \quad 6-2$$

ΔP : change in pressure due to surface tension of water and air

$$\Delta P = 2 \frac{\sigma}{r} \quad 6-3$$

σ : surface tension of water and air (N/m)

By accounting for the change of pressure due to surface tension between air and water, the radii of the compressed air can be determined by solving a cubic equation.

$$Pr^3 + 2\sigma r^2 - PR^3 = 0 \quad 6-4$$

The amount of compressed air in a spherical void can be computed by comparing the initial volume and final volume.

$$\frac{\frac{4}{3}\pi r^3}{\frac{4}{3}\pi R^3} \cdot 100 \quad 6-5$$

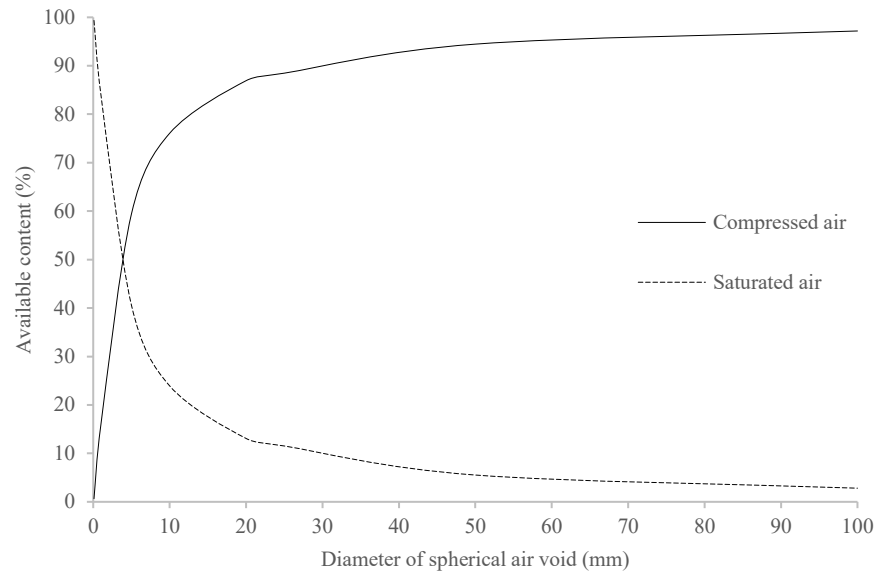


Figure 6-1 Amount of compressed and saturated air in a spherical air void during capillary absorption at 20°C.

6.3 Simulating air voids at the steel-concrete interface

To develop and simulate air voids at the steel-concrete interface numerous methods were considered including artificially imprinting spherical voids. However, it was determined that these methods are not representative of air voids in cementitious materials due to differences in surface morphology and porosity. Thus, affecting the overall microstructure and mass transport properties. This was observed in previous experimental studies conducted by Sandberg (1998), where artificial interfacial voids were developed with filter paper to determine the critical chloride content. It was criticized that filter paper had pores coarser than cement and finer than macroscopic interfacial voids, thus affecting overall results (U. M. Angst et al. 2019). Therefore, an alternative method of injecting air into mortar and cement paste specimens with 1ml syringes during casting was implemented. It was considered that injected air bubbles will retain under poly(methyl methacrylate) sheets and develop entrapped air voids over time.

6.3.1 Methods used to simulate air voids

Mortar and cement paste with a water/cement ratio (w/c) of 0.4 - 0.6 and aggregate volume fraction of 20 -50% were prepared. Cement I 52.5 R was used with the chemical and physical properties specified in Section 3.1.1. Mixes were designed with the proportions shown in Table 6-1.

Table 6-1 Mix proportions.

<i>Mix design</i>	<i>w/b ratio</i>	<i>Water (kg/m³)</i>	<i>Sand (kg/m³) <5mm</i>	<i>Cement (kg/m³)</i>	<i>Aggregate vol fraction: %</i>
M-0.4-PC-20	0.4	436	533.0	1107.1	20
M-0.5-PC-20	0.5	478.4	533.0	972.5	20
M-0.5-PC-30	0.5	421.3	799.0	851.0	30
M-0.5-PC-40	0.5	364.2	1065.0	729.4	40
M-0.5-PC-50	0.5	307.0	1332.0	607.8	50
P-0.4-PC	0.4	554.0	-	1384.0	-
P-0.45-PC	0.45	582.0	-	1294.0	-
P-0.5-PC	0.5	608.0	-	1216.0	-

Four customized 50 × 50 × 50 mm steel moulds with 2 mm diameter holes were drilled at the bottom of base plates with an Axminster ED1682 pillar drill (Figure 6-2 a). After obtaining the customized steel moulds, specimens with the dimensions of 50 × 50 × 50 mm were prepared. Mortar and cement were dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Before casting, drilled holes were sealed with adhesive tape to prevent any leakage. Samples were cast and compacted on a vibrating table until no significant release of air bubbles was observed and the needle was inserted from the bottom to inject specific air volumes of 0.2 and 0.4ml (Figure 6-2 b). Subsequently, moulds were further vibrated for 15 seconds to remove any channelling effects from needle insertion. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of 20±2°C and relative humidity of 95±5% for 2 days. Once the desired curing age has been achieved, the overall volume of air voids was estimated based on the density method by measuring the difference between the final and initial mass of specimens on a scale with an accuracy of ± 0.001g. (Table 6-2). The density of CEM I 52.5R cement was measured with a Micromeritics AccuPyc II gas displacement pycnometer with a displacement medium consisting of nitrogen gas. Subsequently, the diameter and depth of air voids were measured with a vernier calliper with an accuracy of ±0.03 mm.



Figure 6-2 Customized 50 × 50 × 50mm steel mould with a) 2mm diameter drilled holes and b) 1ml syringe with the needle inserted at the bottom of the base plate.

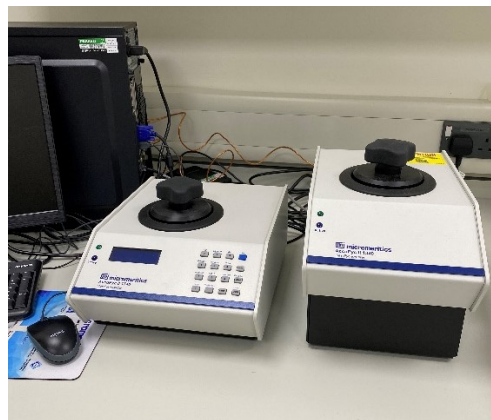


Figure 6-3 Equipment and apparatus setup of Micromeritics AccuPyc II gas displacement pycnometer.

Table 6-2 Geometrical dimensions of air voids.

<i>Sample ID</i>	<i>Void diameter (mm)</i>	<i>Void depth (mm)</i>	<i>Volume of injected air (cm³)</i>	<i>Volume of air void (cm³)</i>
P-0.35-PC	6.5	3.7	0.1	0.03
P-0.45-PC	6.5	3.5	0.1	0.03
P-0.5-PC	6.5	3.5	0.1	0.03
P-0.35-PC	9.5	3.5	0.2	0.07
P-0.45-PC	9.5	3.5	0.2	0.07
P-0.5-PC	9.5	3.7	0.2	0.07
P-0.45-PC	13.0	3.7	0.4	0.12
P-0.5-PC	13.0	3.7	0.4	0.12

From the measurements presented in Table 6-2, it was evident that void diameter, depth and volume were dependent on the amount of injected air. However, entrapped air voids were unable to form on surfaces of mortar specimens with 50% and 60% aggregate volume fractions. This might have resulted from high compression forces exerted by aggregates. Moreover, by comparing the surface morphology of simulated and artificially imprinted air voids, it was discovered that simulated air voids were significantly less porous as shown in Figure 6-4.

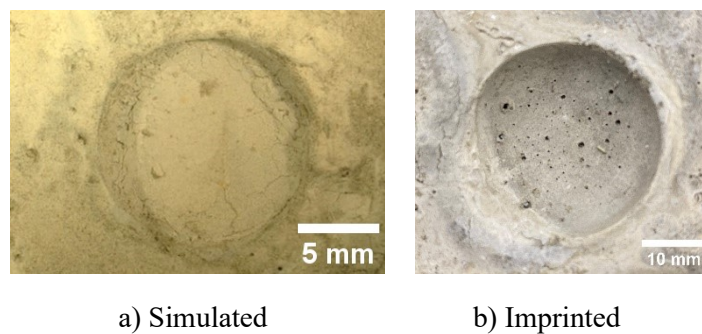


Figure 6-4 Surface morphology of simulated air voids and air voids imprinted with a 20 mm diameter stainless steel spoon on cement paste specimens.

6.3.2 Limitations

Further methods were employed to create air voids with controlled volumes in mortar specimens. This include injecting 0.4 – 0.8 ml of air at 25 mm depth, reducing aggregate volume fraction to 20% and varying vibration times between 1 and 3 minutes. It was considered that a reduction in aggregate volume fraction and an increase in vibration times will decrease compression forces exerted on air bubbles, and encourage spherical voids to form. Nevertheless, after reducing the vertical cover depth to 25mm with an Isomet 1000 precision cutter, it was discovered that voids with conical tips had formed for 20% aggregate volume fraction mortar specimens with 0.4 and 0.6 ml injected air as seen in Figure 6-5. With an increase in vibration time of 1 minute 30 seconds, elongated shapes had formed on surfaces (Figure 6-6). Similarly, air voids with elongated shapes and conical tips were entrapped in mortar specimens with a 30% aggregate volume fraction (Figure 6-7). Moreover, no voids were found in mortar specimens with a

50% aggregate volume fraction (Figure 6-8). Thus, after multiple trials, it was determined that air voids with identical geometrical dimensions cannot be replicated in mortar specimens.

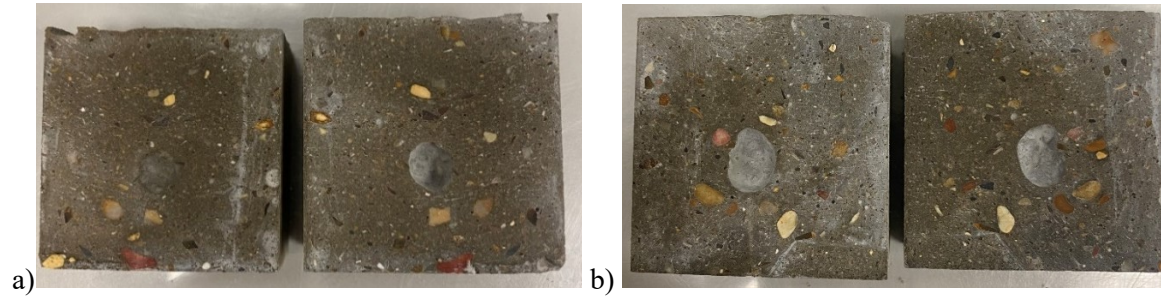


Figure 6-5 Cross-sectional view of 20% aggregate volume and 0.4 w/c mortar samples with a) 0.4 ml and b) 0.6 ml of air injected after 1 minute 30 seconds of vibration.

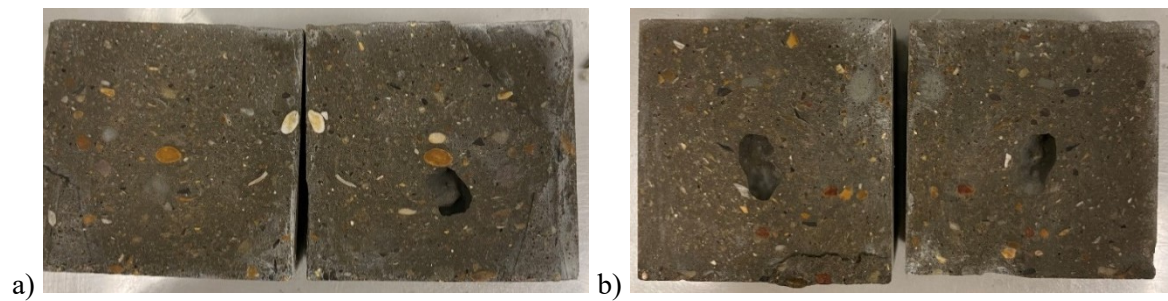


Figure 6-6 Cross-sectional view of 20% aggregate volume and 0.4 w/c mortar samples with a) 0.4 ml and b) 0.6 ml of air injected after 3 minutes of vibration.

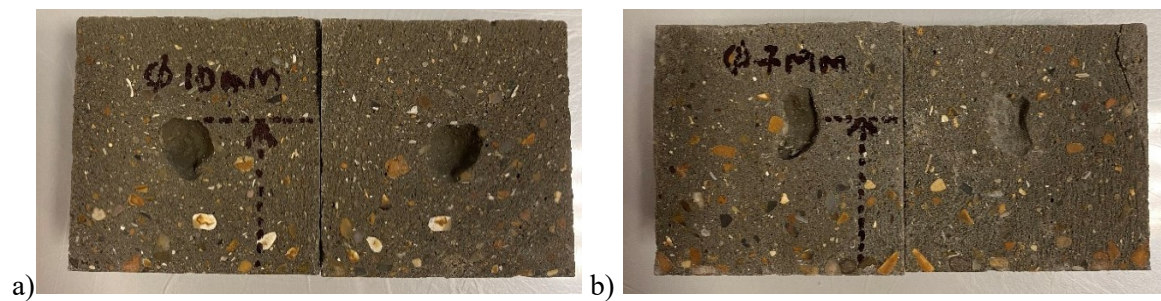


Figure 6-7 Cross-section view of 30% aggregate volume and 0.5 w/c mortar samples with a) 0.6 ml and b) 0.8 ml of injected air after 1 minute 30 seconds of vibration.



Figure 6-8 Cross-section view of 50% aggregate volume mortar sample with 0.6 ml of injected air after 1 minute of vibration.

6.3.3 Influence on transport properties

The impact of channelling effects on transport properties was further investigated with fluorescence imaging and gravimetric analysis. This was done to ensure that the developed method for simulating air voids in cement paste specimens will not affect the transport of moisture.

6.3.3.1 Samples

Specimens with the dimensions of $50 \times 50 \times 50$ mm were cast with the mix proportions shown in Table 6-1. Cement was dry mixed in a pan mixer for 30 seconds and wet mixed for 3 minutes after the addition of water. Samples were cast and compacted on a vibrating table until no significant release of air bubbles was observed and needles were inserted at the corners of steel moulds to replicate channelling effects. Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $95 \pm 5\%$ for 2 days. In addition, the cover depth of selected samples was reduced to 25mm with Isomet 1000 precision cutter and conditioned in 50°C oven for four weeks till a mass change smaller than 0.01% was obtained.

6.3.3.2 Fluorescence imaging technique

After curing specimens with and without needle insertion for 3 days, the exposed surface was placed on two 5×5 mm plastic rods in a petri dish containing 1% wt. fluorescein water at a depth of 6-7 mm (Figure 6-9). Each sample was submerged for 5 and 20 minutes, and images were captured with a stereo microscope Olympus SZX10 under ultraviolet light. Throughout the test, care was taken to ensure that the solution level was maintained.

6.3.3.3 Water absorption

Prior to the water sorptivity test, specimens with a 25 mm cover depth were wrapped in cling film and placed in polyethylene bags to cool for at least 12 hours till a mass change smaller than 0.02% was obtained. The exposed surface was placed on two 5×5 mm plastic rods in a container containing shallow water at a depth of 6-7mm. Masses of each sample were measured at 10, 15, 20 and 30 minutes, then at 15-minute intervals for the next hour and every hour for the next 6 hours by which time saturation had been achieved. Throughout the test, the container was covered with a lid and weighing was carried out as rapidly as possible to minimise water evaporation. Care was taken to ensure that the water level was maintained.

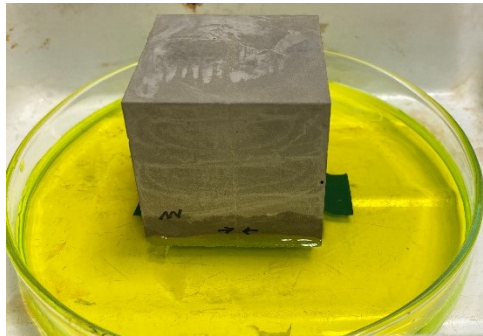


Figure 6-9 Exposing cement paste specimen to water containing 1% wt. fluorescein.

6.3.3.4 Result and discussion

From the images presented in Figure 6-10, channelling effects from needle insertion can be observed. A pointed curvature shape was formed in specimens without vibration including regions with higher colour contrast located away from the insertion site. This can be resulted of the compaction of cement particles with a variation in porosity and uneven distribution of fluorescein across samples. These effects were more prominent at higher water to cement ratios with areas of higher colour contrast (Figures 6-12 and 6-14). On the contrary, an even distribution of fluorescein was observed by vibrating specimens after needle insertion (Figure 6-11). This indicates that the effects caused by channelling were mitigated. In addition, the effects of channelling were less evident for 0.5 w/c cement paste specimens (Figures 6-16 and 6-17). This is because cement particles were able to disperse evenly in mediums with lower viscosity.

Moreover, near identical rates of absorption were measured for specimens with and without needle insertion (Figure 6-18). These results suggest that the transport of moisture was unaffected by revibrating samples after needle insertion, and are in agreement with images obtained from fluorescence imaging.

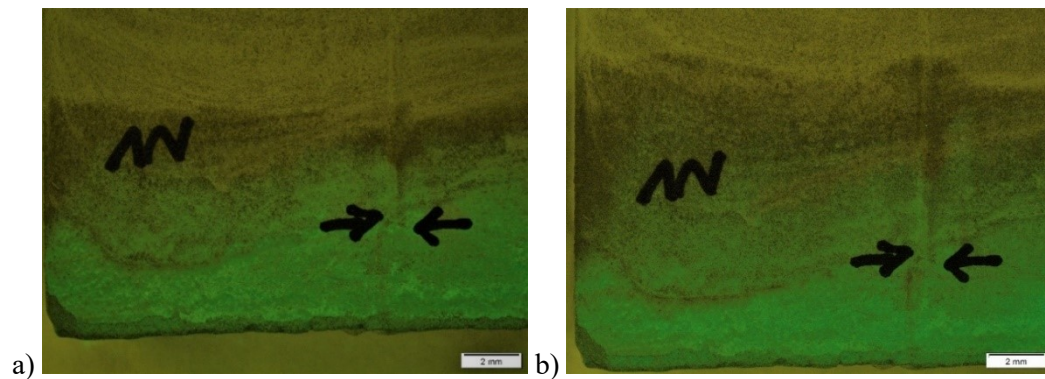


Figure 6-10 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.35 w/c cement paste specimens without vibration.

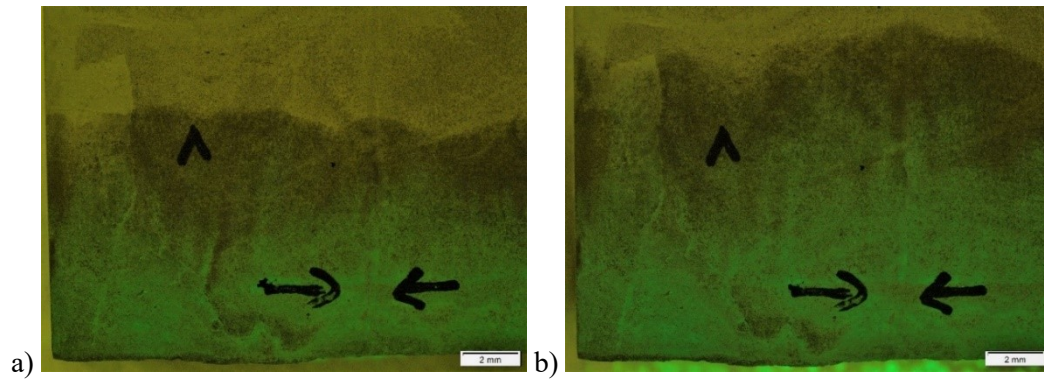


Figure 6-11 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.35 w/c cement paste specimens with vibration.

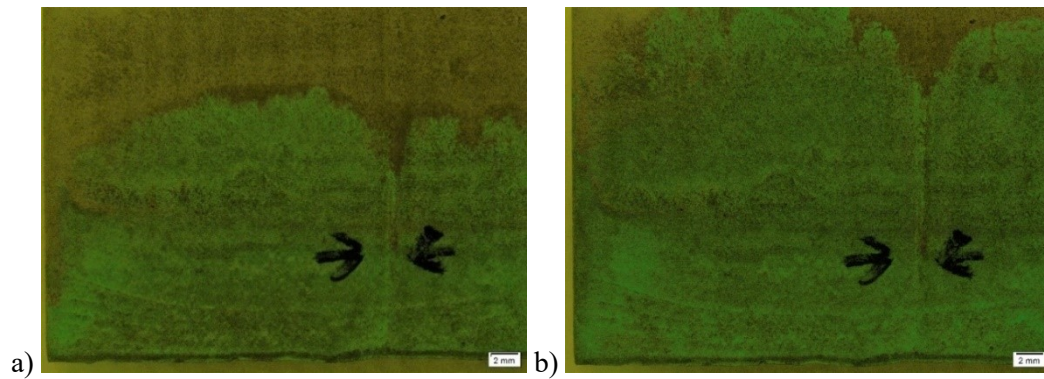


Figure 6-12 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.4 w/c cement paste specimens without vibration.

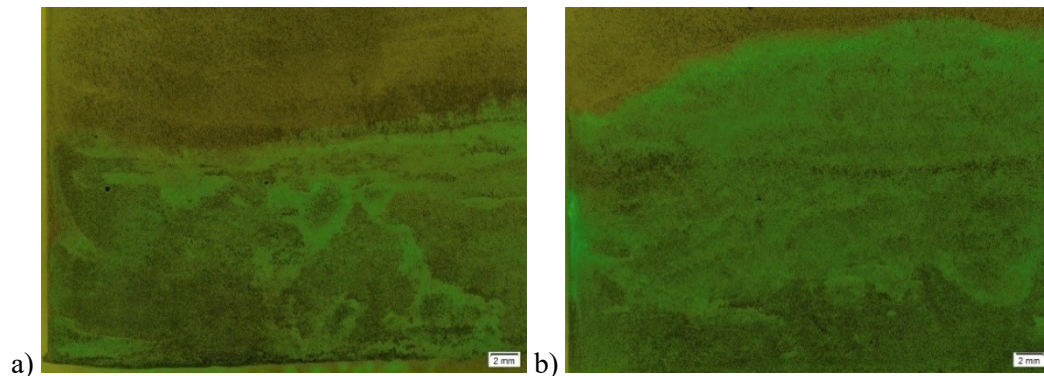


Figure 6-13 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.4 w/c cement paste specimens with vibration.

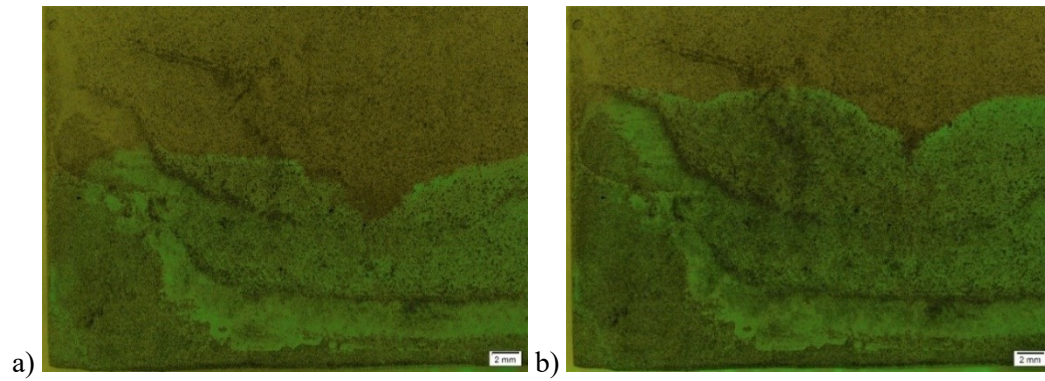


Figure 6-14 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.45 w/c cement paste specimens without vibration.

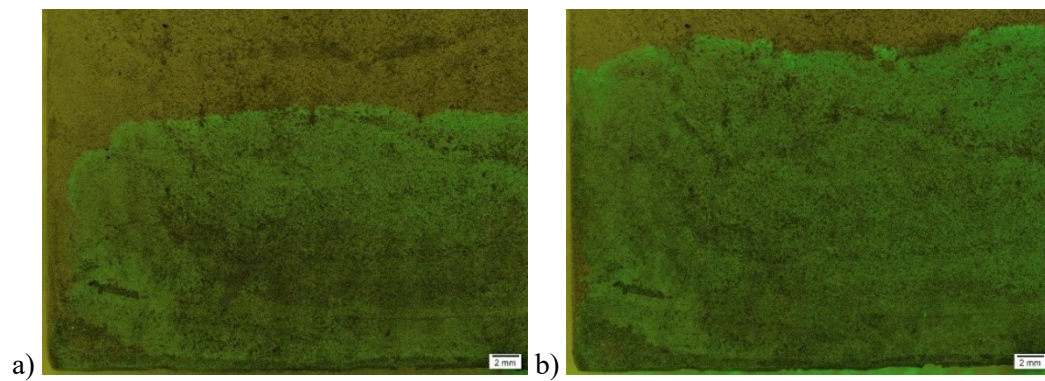


Figure 6-15 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.45 w/c cement paste specimens with vibration.

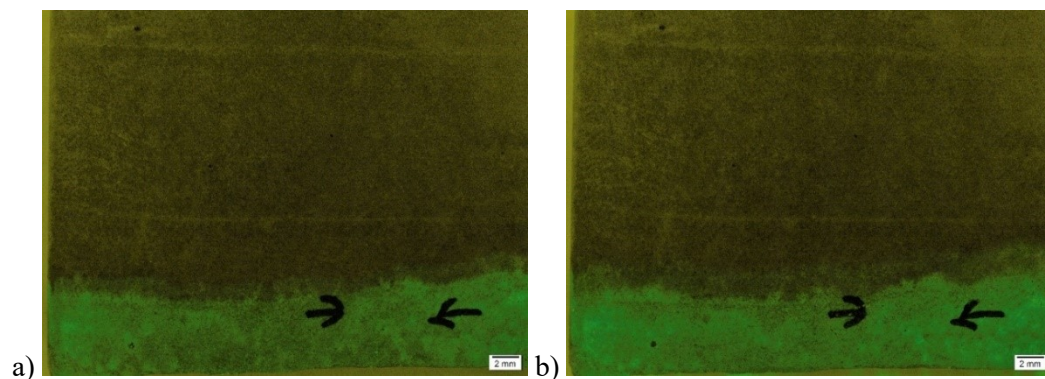


Figure 6-16 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.5 w/c cement paste specimens without vibration.

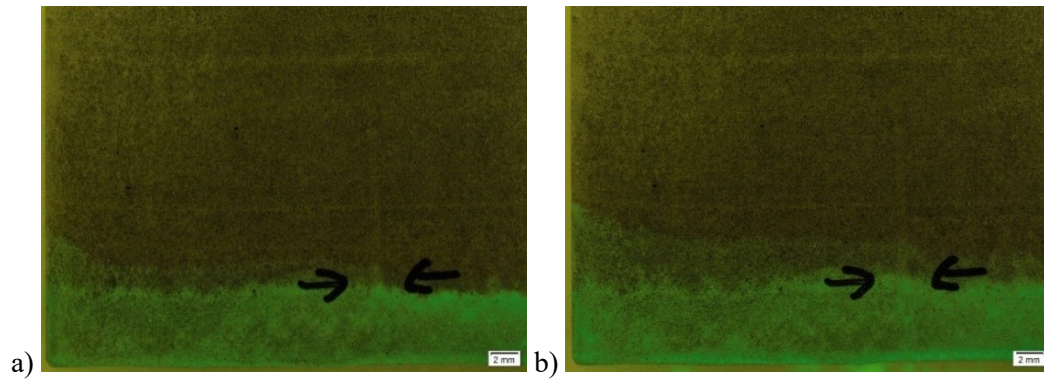


Figure 6-17 Images captured with a stereo microscope Olympus SZX10 after a) 5 minutes and b) 20 minutes of capillary absorption in water containing 1% wt. fluorescein illuminated with UV light for 0.5 w/c cement paste specimens with vibration.

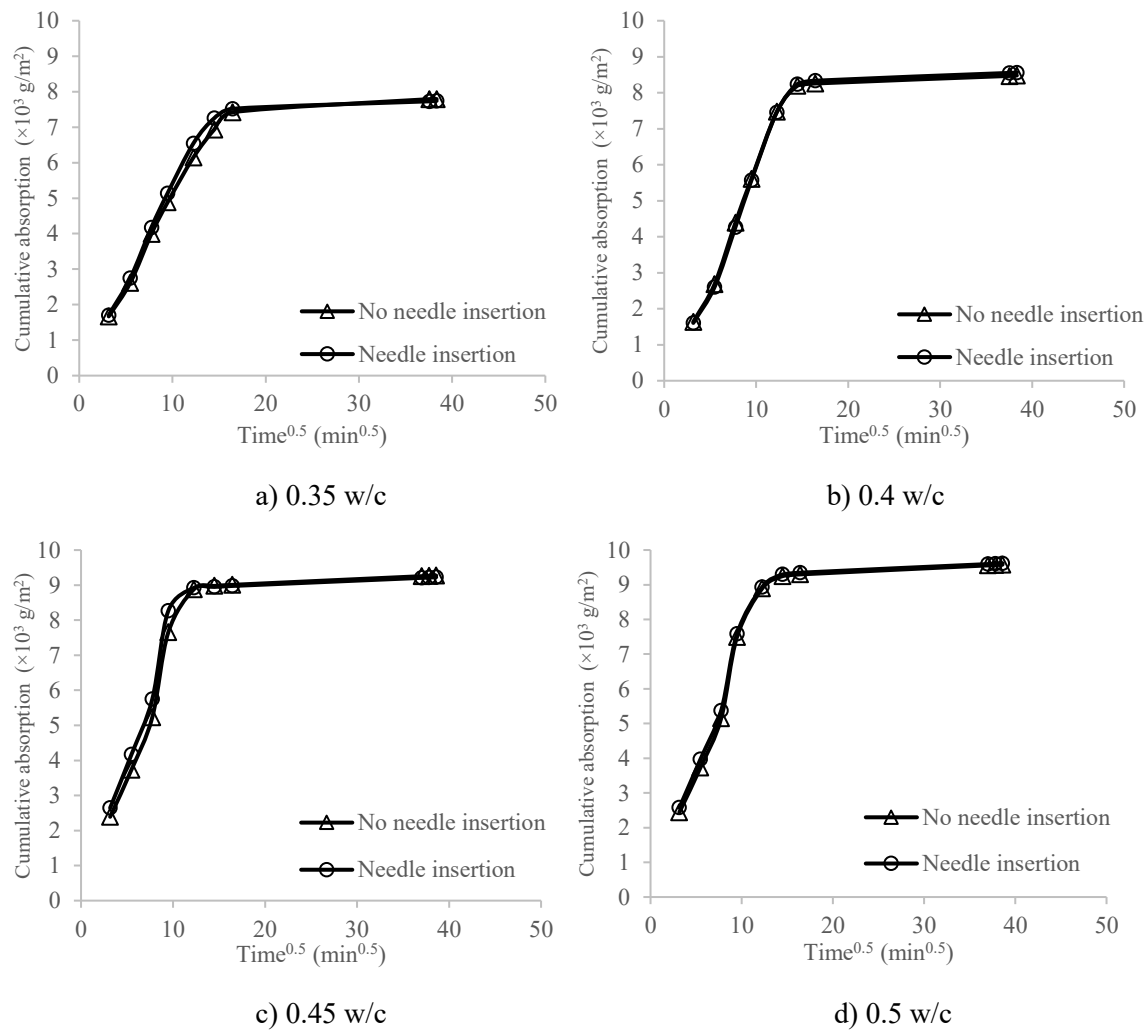


Figure 6-18 Cumulative absorption of 3 day cured 0.35w/c, 0.4 w/c, 0.45 w/c and 0.5 w/c cement paste specimens with needle insertion and without insertion.

6.3.3.5 Summary

The method used to develop air voids in cementitious material including limitations and influence on transport properties was described. It was shown for the first time that air voids with near identical diameter, depth and volume can be created in cement paste specimens. However, after multiple trials, air voids with controlled volumes cannot be replicated in mortar specimens with aggregate volume fractions ranging from 20- 60%. This has resulted from compression forces exerted by aggregates. The influence of needle insertion on transport properties was verified by fluorescence imaging and gravimetric analysis. Images and results obtained demonstrated that the transport of moisture and fluorescein particles was not affected by revibrating specimens.

6.4 Experimental

6.4.1 Materials and mix proportions

Cement paste with a water/cement ratio (w/c) of 0.35 - 0.5 was prepared. Cement I 52.5 R was used with the chemical and physical properties specified in Section 3.1.1. Mixes were designed with the proportions shown in Table 6-3.

Table 6-3 Mix proportions.

<i>Mix design</i>	<i>w/b ratio</i>	<i>Water (kg/m³)</i>	<i>Cement (kg/m³)</i>
P-0.35-PC	0.35	520.4	1486.8
P-0.45-PC	0.45	582.5	1294.3
P-0.5-PC	0.5	607.8	1215.7

6.4.2 Samples

Sixty-six cement paste specimens with the dimensions of 50 × 50 × 50 mm were prepared. Cement paste was batched in a mixer and cast in steel moulds. Before casting, drilled holes were sealed with adhesive tape to prevent any leakage. Samples were compacted on a vibrating table until no significant release of air bubbles was observed. Subsequently, the top surface of the steel mould was covered with a 5mm thick

Perspex sheet and a needle was inserted from the bottom through 2mm diameter drilled holes to inject specific air volumes of 0.1, 0.2 and 0.4ml. After injection, samples were revibrated for 15 seconds to remove any channelling effects caused by needle insertion.

Compacted samples were covered with wet hessian fabric at room temperature for 24 hours to prevent drying. After 24 hours, samples were removed from the moulds and placed in a curing room at a temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $95 \pm 5\%$ for 3, 14 and 28 days. Once these curing ages have been reached, the horizontal cover depth was reduced to 25mm with Isomet 1000 precision cutter. Selected specimens were placed in an oven at 50°C , and incubators containing sodium chloride and potassium dichromate salt solutions at 55% and 75% RH in a controlled temperature laboratory at $21 \pm 2^\circ\text{C}$. The mass of each sample was measured for two months until a change smaller than 0.01% was obtained.

6.4.3 Water absorption

To characterize the ability of specimens to absorb and transport water, a conventional gravimetric measurement was used to monitor the change of mass over time at ambient temperature ($21 \pm 2^\circ\text{C}$). Prior to water sorptivity tests, specimens conditioned in 50°C oven were wrapped in cling film and placed in polyethylene bags to cool for at least 12 hours until a mass change smaller than 0.02 % was obtained. Subsequently, the sides were coated with epoxy resin to allow unidirectional capillary absorption. The exposed surface was placed on two 5×5 mm plastic rods in a container containing shallow water at a depth of 6-7 mm (Figure 6-19). The mass of each sample was measured at 10, 15, 20 and 30 minutes, then at 15 minute intervals for the next hour and then every hour for the next 6 hours by which time saturation had been achieved. Throughout the test, the container was covered with a lid and weighing was carried out as rapidly as possible to minimise water evaporation. Care was taken to ensure that the water level was maintained.

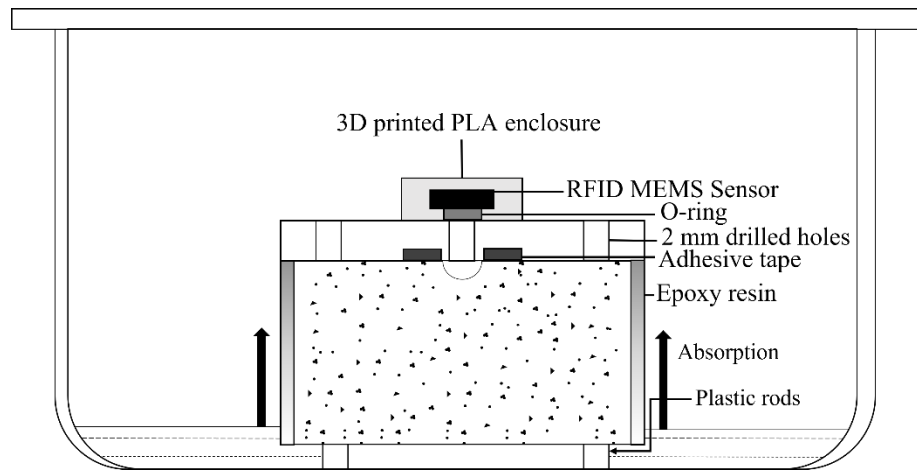


Figure 6-19 Schematics of experimental setup for water absorption.

6.4.4 Water desorption

The capability of specimens to retain moisture in relation to the reduction of relative humidity in air voids was quantified and analyzed by conducting water desorption tests. The top surface of specimens was sealed with adhesive tape and placed on two 5×5 mm plastic rods to allow unidirectional capillary desorption (Figure 6-20). Masses of each sample were measured at 60, 120 and 180 minutes, then at 24 hour intervals for the next 6 days till a mass change of smaller than 0.01% was obtained.

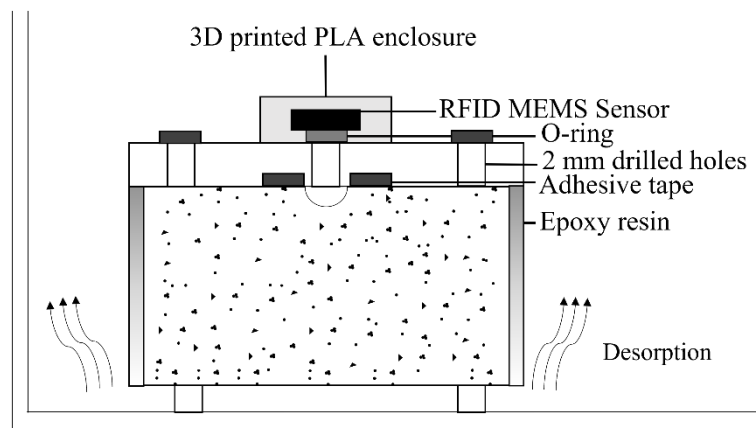


Figure 6-20 Schematics of experimental setup for water desorption.

6.4.5 Monitoring internal relative humidity

The internal relative humidity of air voids during water absorption and desorption was monitored with RFID MEMS sensors and reader (Figure 6-22). To provide a direct contact and proper seal between the MEMS sensor and air void, $43 \times 43 \times 15$ mm PLA enclosures were 3D printed and bonded onto a 2mm thick Perspex sheet (Figure 6-21). In addition, a nitrile rubber o-ring and adhesive tape were placed around MEMS sensor and entrapped air void to prevent any leakages. To ensure that saturated air does not seep into the designed experimental setup, relative humidity was monitored in 100% relative humidity environment and laboratory conditions for 12 hours. Near identical relative humidity measurements were obtained indicating that saturated air does not seep into the designed experimental setup (Figure 6-23).

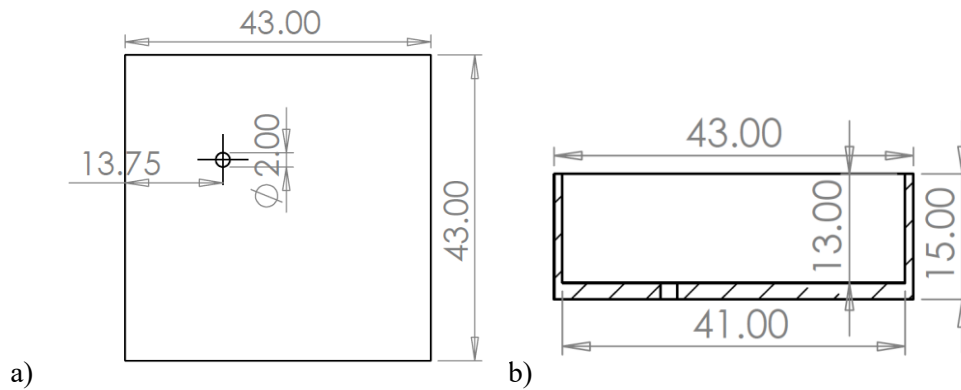


Figure 6-21 Schematics of 3D printed PLA enclosure with a) top and b) side view.

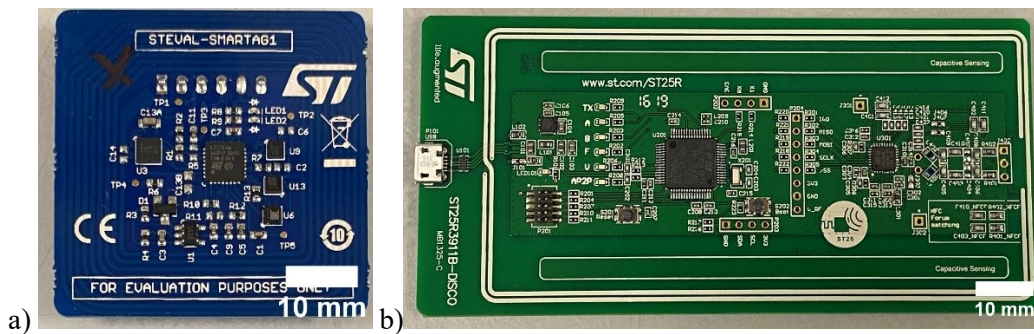


Figure 6-22 Images of a) STMicroelectronics STEVAL SMARTAG1 and b) ST25R3911B-DISCO RFID reader.

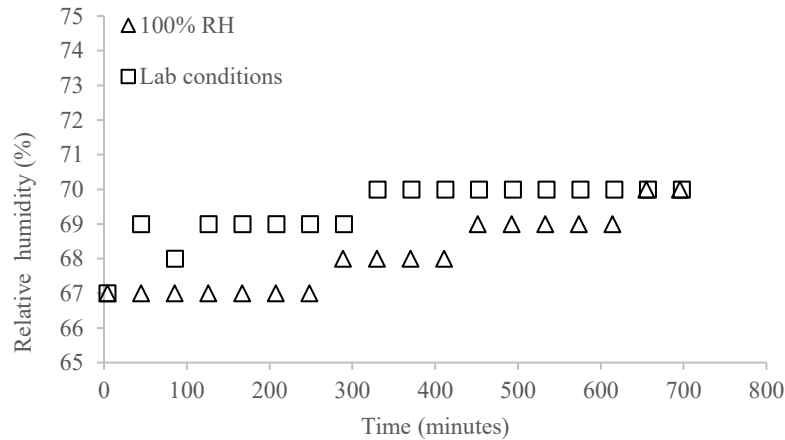


Figure 6-23 Testing leakage of developed technique to monitor the internal relative humidity of air voids.

6.4.6 Olympus SZX10 stereomicroscopy

The surface morphology of air voids was examined after curing and exposure to water absorption and desorption tests. This was done by placing specimens under an Olympus SZX10 stereomicroscope with the equipment and setup shown in Section 3.3.3. Images were captured and further processed with Fiji (Schindelin et al. 2012) by manually tracing surface cracks. To obtain accurate measurements, each image was calibrated with known distances and pixel aspect ratio. 15 measurements were taken for each crack length with the overall average value presented.

6.4.7 Backscattered electron microscopy (BSE)

Cracks at the paste-void interface were investigated by encasing the sides of specimens with epoxy resin in a mould with the dimensions of $65 \times 65 \times 30$ mm after water desorption tests. Subsequently, samples were vacuum impregnated with epoxy resin, cut and grind with the steps and equipment described in Section 3.2.2.1. Samples were examined at $\times 25$ magnification with a Hitachi TM4000 Plus scanning electron microscope (SEM) shown in Section 3.2 with a 5 mm spacing between each segment (Figure 6-24).

6.4.7.1 Analysis

Images captured were imported into Fiji (ImageJ version 1.53t NIH) with individual cracks manually traced. Each image was calibrated with known distances and pixel aspect ratio to obtain accurate measurements. A total of 15 measurements were obtained for each crack length to account for errors with the overall average value presented.

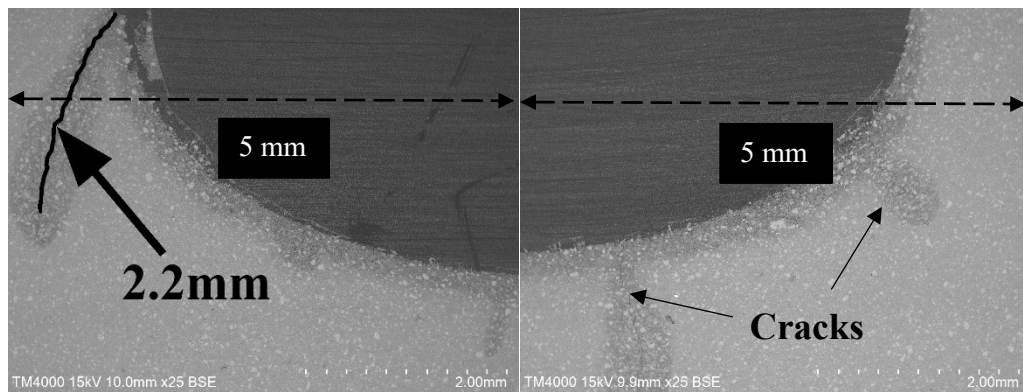


Figure 6-24 Examples of microcracks at the paste-void interface examined with SEM/BSE after conditioning at 50°C and exposure to wetting and drying.

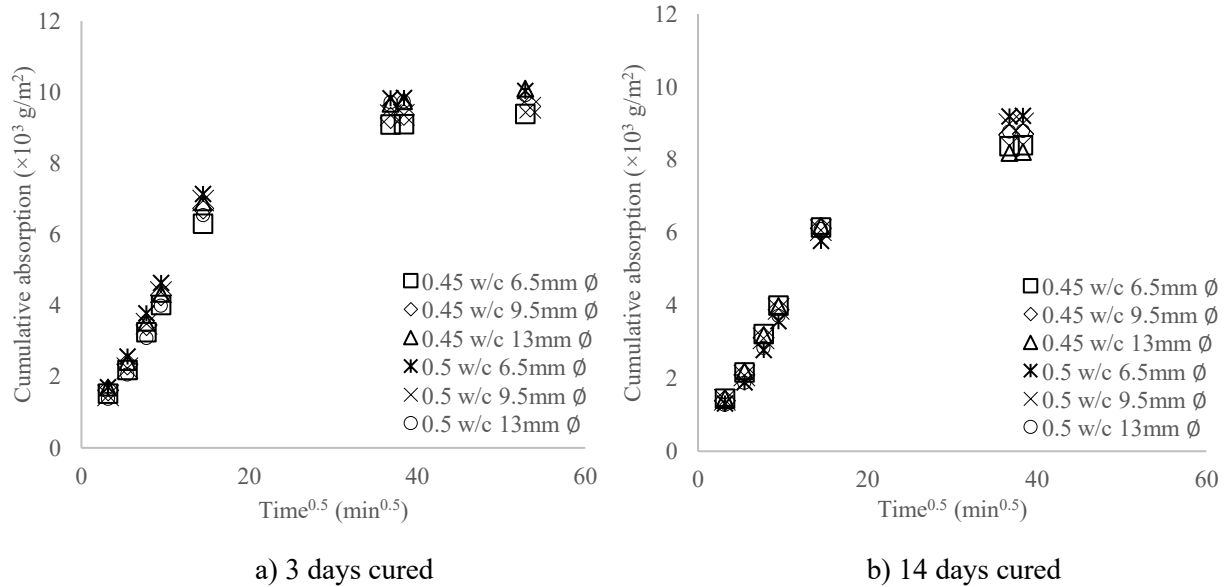
6.5 Results

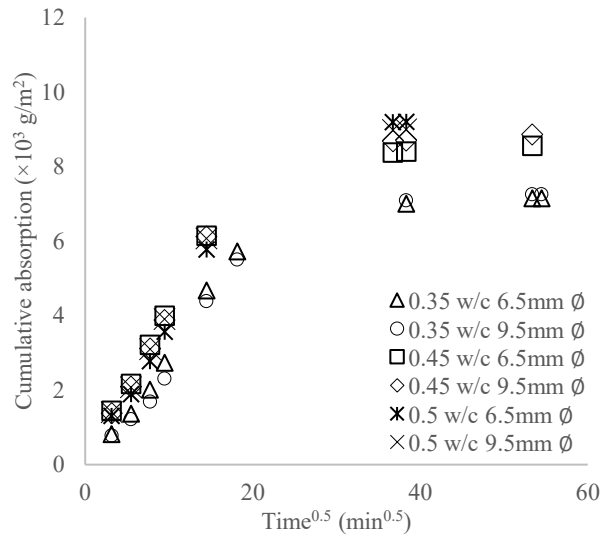
6.5.1 Cumulative absorption vs. $t^{1/2}$ plots

Figures 6-25 and 6-26 present examples of cumulative absorption (mass per unit inflow surface area) against square-root of elapsed time ($\sqrt{t}$) for cement paste specimens of various water to cement ratios, dimensions of air voids, curing age and conditioning environments. Replicate samples produced near identical water absorption characteristics and thus, were not presented for clarity.

From the results obtained, it was evident that specimens with lower water to cement ratio and greater curing age had an overall decrease in sorptivity and total absorption. Cumulative absorption for cement

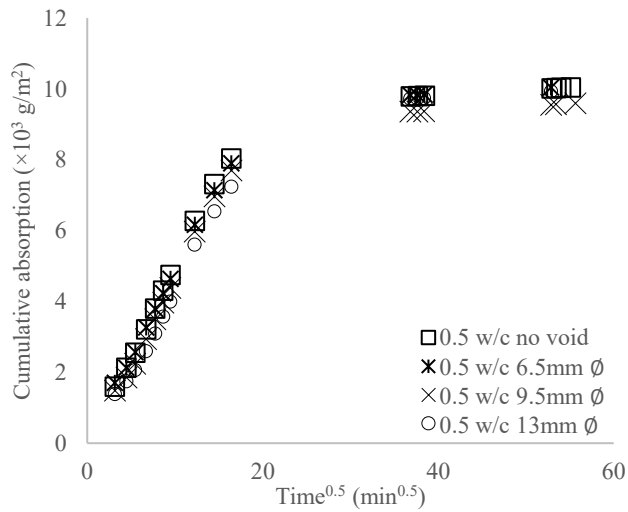
paste samples cured for 3 days with 0.5 w/c and 9.5 mm diameter air void started to equilibrate at approximately $10 \times 10^3 \text{ g/m}^2$. In comparison, 0.35 and 0.5 w/c cement paste samples cured for 28 days with identical void diameters of 9.5 mm equilibrated at $7.3 \times 10^3 \text{ g/m}^2$ and $9 \times 10^3 \text{ g/m}^2$ (Figure 6-25). These observations were expected because water absorption is dependent on capillary porosity. Samples with a lower abundance of empty pores have a lower capillary suction and absorption (Nokken and Hooton 2002). Additionally, it was interesting to note that specimens with air voids of different dimensions had near identical rates of cumulative absorption. This is because specimens with identical water to cement ratio and curing age will have similar bulk volume and porosity. Nevertheless, there was a slight increase in cumulative absorption at 0 to 20 $\text{min}^{0.5}$ for 0.5 w/c cement paste specimen with 9.5 mm diameter void (Figure 6-26 c). This can be attributed to cracks present at the paste-void interface. On the other hand, a greater total absorption was measured for specimens conditioned at 50°C compared to samples conditioned at 55% and 75% RH. When conditioning samples at 50°C most capillary pores will be completely dried and additional microcracks can be induced (Wu et al. 2019).



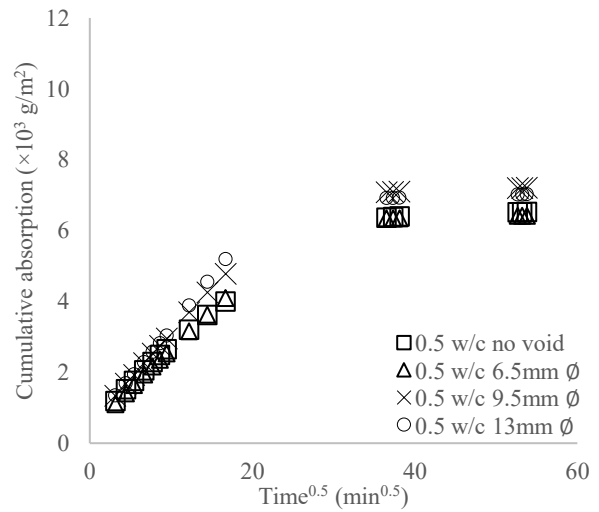


c) 28 days cured

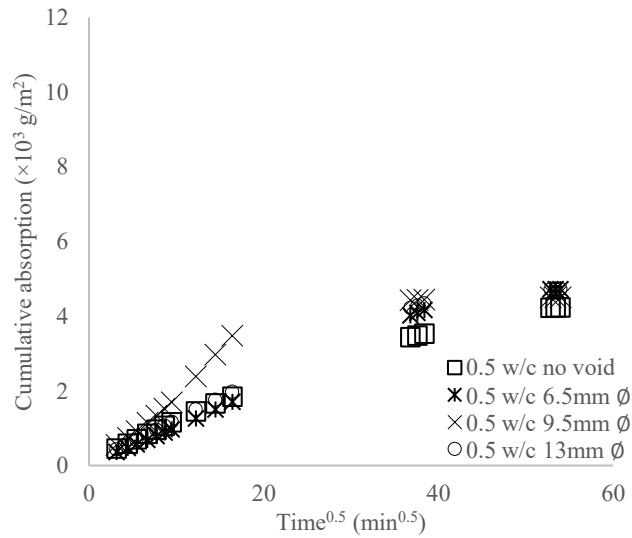
Figure 6-25 Examples of cumulative water absorption plots for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C.



a) 50°C



b) 55% RH



c) 75% RH

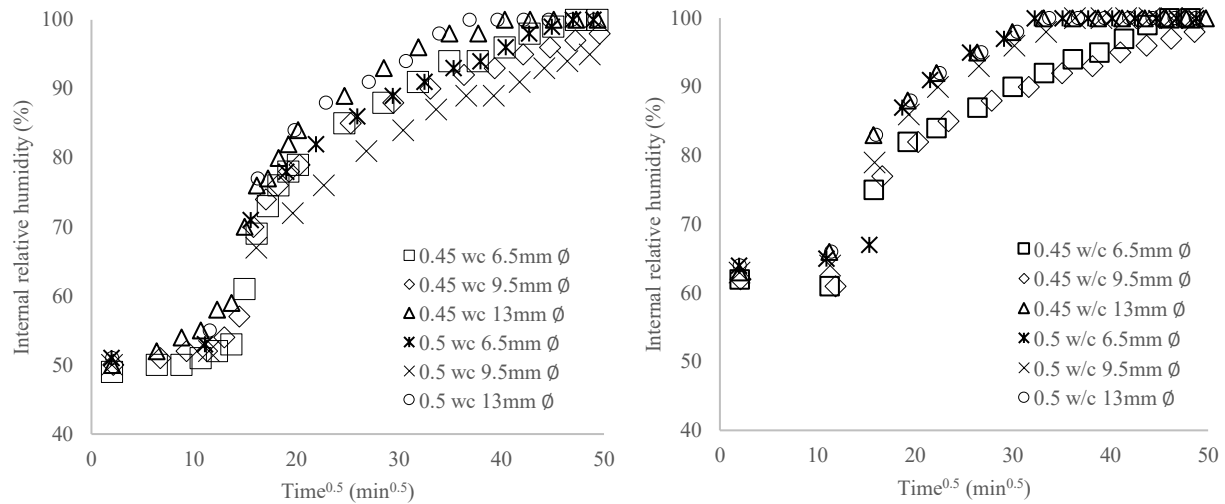
Figure 6-26 Examples of cumulative water absorption plots for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.

6.5.2 Rate of increase in relative humidity

Figures 6-27 and 6-28 plot the increase of internal relative humidity against the square-root of elapsed time ($\sqrt{t}$) to examine the influence of the geometrical size of air voids, water to cement ratio, curing age and conditioning environments. To produce these plots, samples of identical curing age and water to cement ratio are sorted into groups. The overall trend suggests that air voids with a diameter of 13mm saturated at a faster rate compared to 9.5mm (Figure 6-27). Nonetheless, a different observation was found for air voids with diameters smaller than 9.5 mm. Air voids with a diameter of 6.5mm saturated at a much faster rate compared to 9.5mm. It was expected because moisture can saturate a smaller void space faster than a larger void space. This is in agreement with specimens conditioned at 55% and 75% RH with faster rates of saturation for smaller air voids (Figure 6-28).

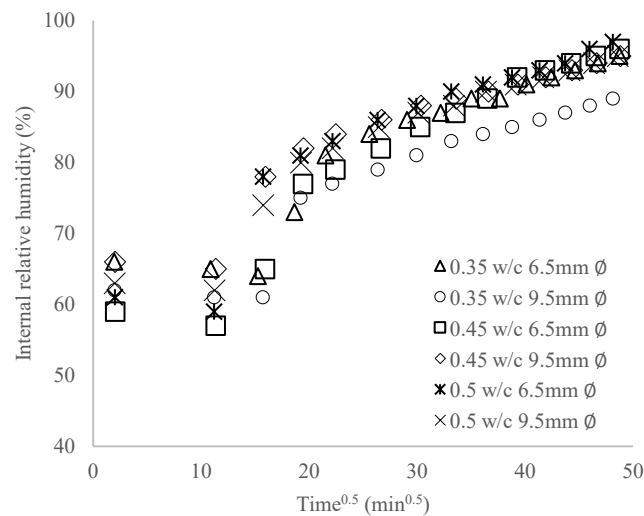
Similarly, a decrease in water to cement ratio and an increase in curing age prolongs the time required to reach 100% RH. This is attributed to a reduction in accessible porosity and the formation of hydration products when cement ages. In addition, it was interesting to note that relative humidity increased at a faster rate for samples without air voids. However, contradictory results were obtained for samples gently dried at 75% RH where air voids saturated at the fastest rate. This can be contributed by moisture present

in capillary pores and surfaces before conducting water absorption experiments. Moreover, by comparing cumulative water absorption plots with internal relative humidity measurements it was evident that relative humidity increased before samples achieve saturation.



a) 3 days cured

b) 14 days cured



c) 28 days cured

Figure 6-27 Examples of internal relative humidity measurements for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C.

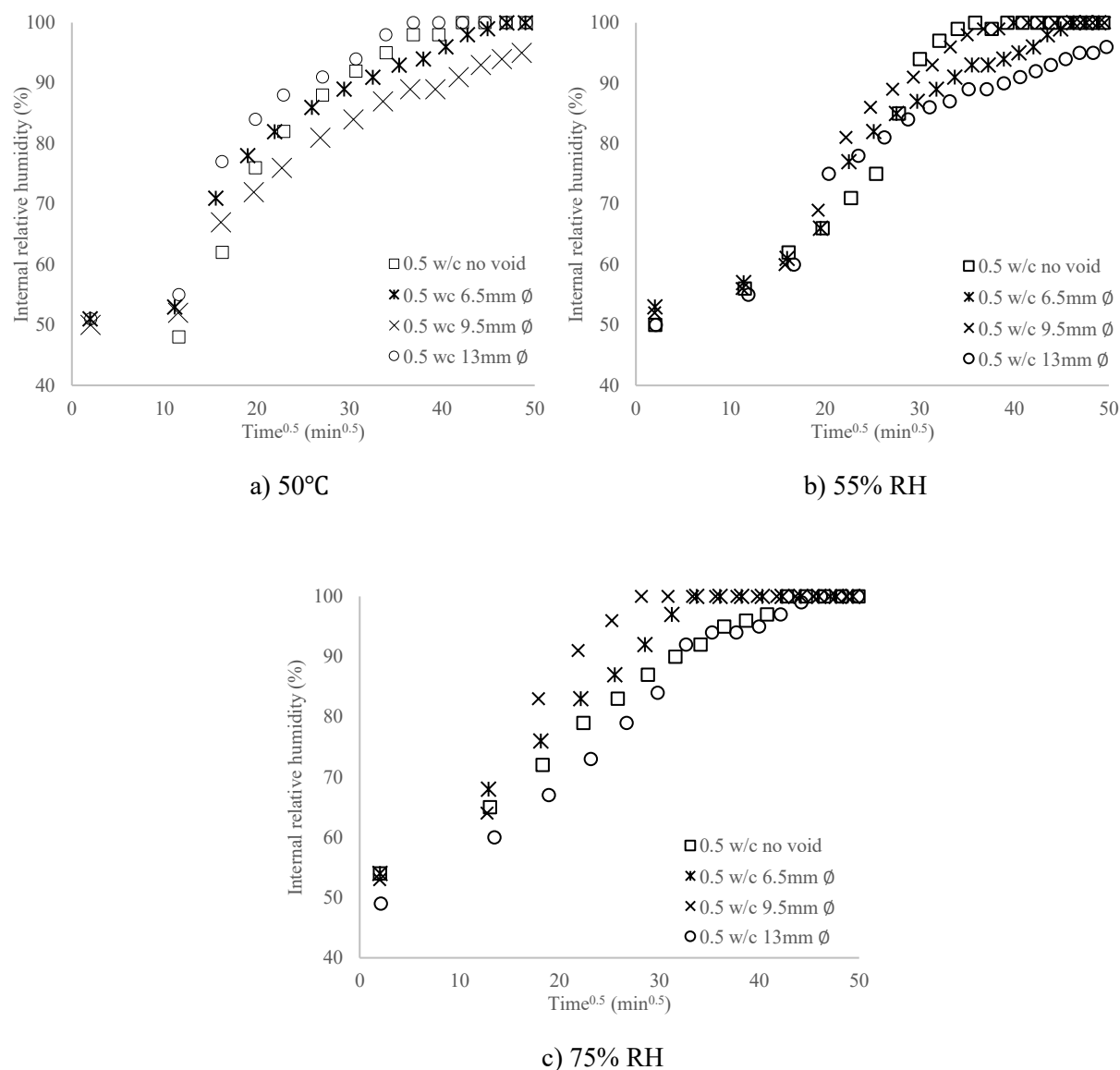


Figure 6-28 Examples of internal relative humidity measurements for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.

6.5.3 Cumulative desorption

Figures 6-29 and 6-30 present examples of cumulative desorption (mass per unit outflow surface area) against square-root of elapsed time ($\sqrt{t}$) for cement paste specimens of various water to cement ratios, dimensions of air voids, curing age and conditioning environments. Replicated samples produced near identical water desorption characteristics; hence, were not presented for clarity.

The obtained gravimetric measurements revealed that desorption occurs at a much faster rate for samples with a greater curing age. Total desorption at $92 \text{ min}^{0.5}$ for cement paste sample cured for 3 days with 0.45 w/c and 9.5 mm diameter air void was approximately $1.6 \times 10^3 \text{ g/m}^2$. As opposed to 0.45 w/c cement paste sample cured for 14 days with a void diameter of 9.5 mm at $2.11 \times 10^3 \text{ g/m}^2$ (Figure 6-29). This was expected because specimens with greater curing age had a lower moisture content. Furthermore, it was interesting to note that specimens with air voids of different dimensions had near identical rate of cumulative desorption. This was similar to the results presented in section 6.5.1. However, there was a significant increase in desorption for 0.5 w/c cement paste specimen with 6.5 and 9.5 mm diameter void conditioned at 50°C and 75% RH (Figure 6-30).

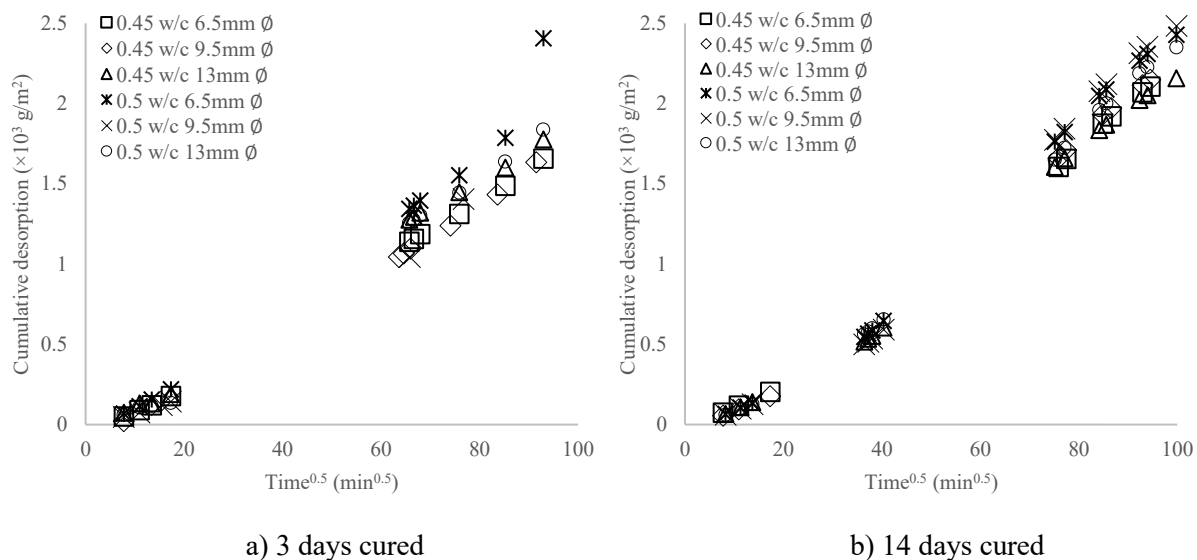


Figure 6-29 Examples of cumulative water desorption plots for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C .

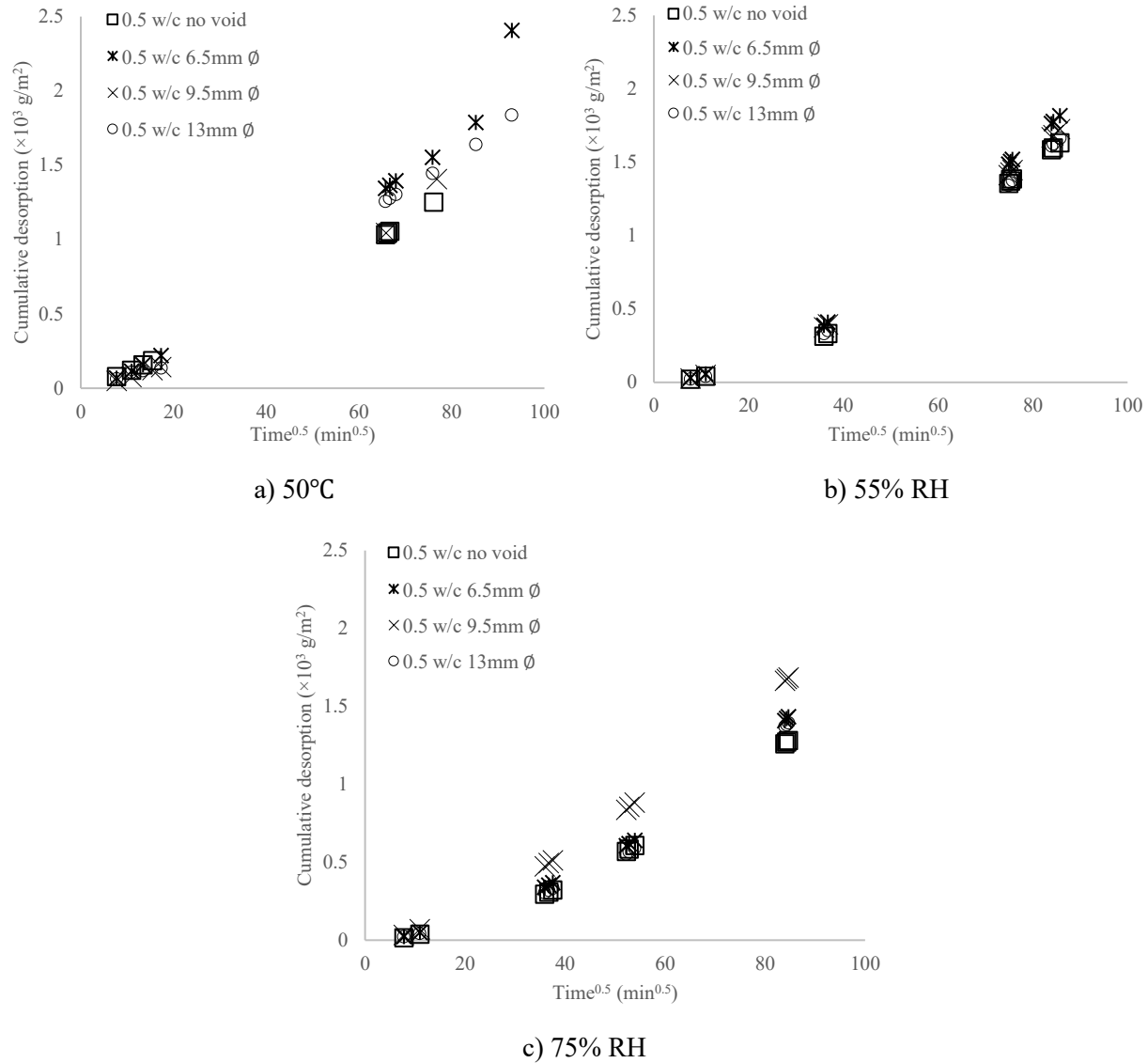


Figure 6-30 Examples of cumulative water desorption plots for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.

6.5.4 Rate of decrease in relative humidity

Figures 6-31 and 6-32 plot the rate of decrease in internal relative humidity against the square-root of elapsed time ($\sqrt{t}$) to examine the influence of the geometrical size of air voids, water to cement ratio, curing age and conditioning environments. To develop a clear analysis, results with identical curing age and water to cement ratio were sorted into groups.

Data obtained show a slower decrease in relative humidity for specimens with greater curing age. A relative humidity measurement of 78% was obtained at $29 \text{ min}^{0.5}$ for 0.45 w/c cement paste samples cured for 3 days with 13 mm diameter air void. In contrast, a relative humidity measurement of 83% was obtained at $29 \text{ min}^{0.5}$ for 0.45 w/c cement paste samples cured for 14 days with 13 mm diameter air void. This was an interesting observation, despite the higher desorption rates measured from gravimetric analysis presented in section 6.3.3. This indicates that moisture will preferably diffuse out of voids from samples with a higher porosity. On the other hand, relative humidity decreased at similar rates for samples with and without voids. This is in agreement with the findings discussed in section 6.3.3, where a near identical cumulative desorption rate was discovered. Additionally, the overall trend suggests that larger voids will desaturate at a faster rate compared to smaller voids. Nevertheless, there was a slower decrease in relative humidity for 0.5 w/c cement paste specimen with 9.5 mm diameter void conditioned at 50 °C, 55% and 75% RH (Figure 6-32).

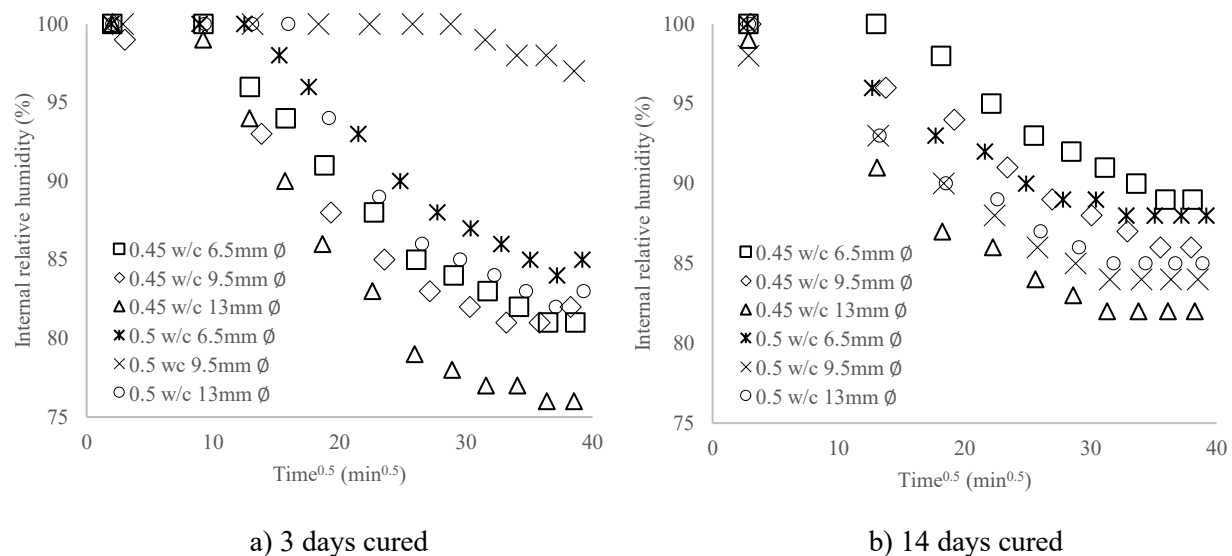


Figure 6-31 Examples of internal relative humidity measurements for 25mm thick cement paste specimens with various diameter entrapped air voids and water to cement ratios conditioned at 50°C.

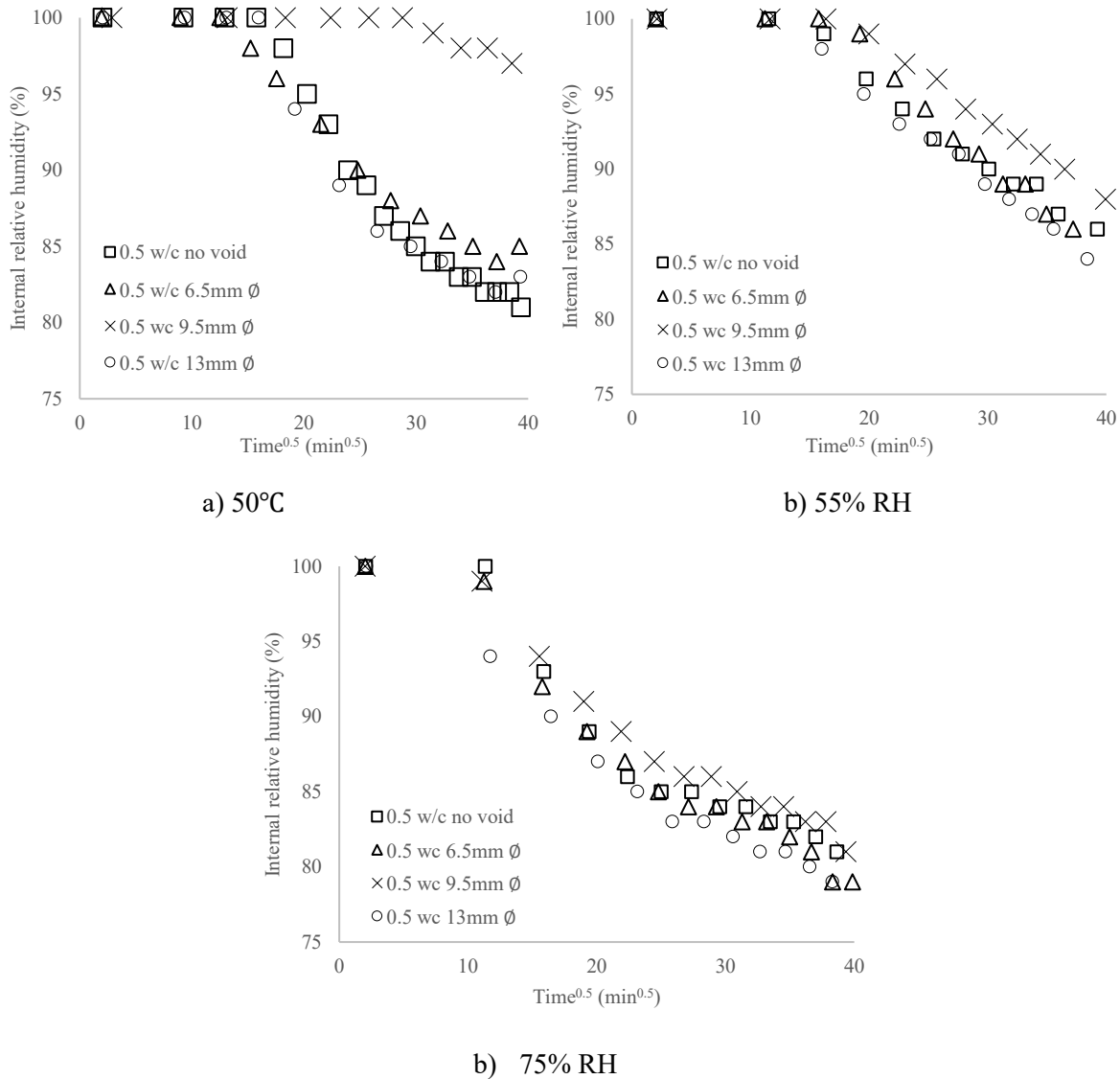


Figure 6-32 Examples of internal relative humidity measurements for 25mm thick 3 days cured cement paste specimens with and without entrapped air voids.

6.5.5 Surface morphology of air voids

Figures 6-33 to 6-44 present surface morphology images of air voids after curing and exposure to wetting and drying to investigate the influence of the geometrical size of air voids, water to cement ratio and conditioning environments on induced cracks. To enhance clarity and quantification, images were captured with identical magnification and exposure settings. Additionally, samples with identical water to cement ratio and conditioning environments were sorted into groups.

From the images captured, it was observed that the majority of samples had small amounts of microcracks on surfaces. It was also evident that the length of microcracks intensifies with higher water to cement ratio (Figures 6-33 and 6-35). Cement paste sample with 0.45 w/c and 9.5 mm diameter air void had a surface crack of 8.8mm. In comparison, cement paste sample with 0.5 w/c and 9.5 mm diameter air void had a surface crack of 10.8mm. This is attributed to the reduction in compressive strength causing cement paste surrounding an air void to collapse when setting time has been achieved. Similar findings were observed for air voids with larger diameters with an overall increase in crack length. Larger air bubbles can potentially collapse much earlier during the curing stage due to the inability to withstand high surface tension forces when cement paste hardens. Microcracks formed are mostly resulted from shrinkage and an increase in tensile strain capacity when moisture is loss when cement hardens (Wang et al. 2013; Wittmann 1968). Additionally, it was interesting to observe similar quantities of cracks on surfaces of air voids after subjecting to wetting and drying and different drying conditions. However, by examining cracks at the paste-void interface shorter cracks were formed during gentle drying compared to more severe drying. A crack length of 4.5mm was measured for 0.5 w/c cement paste sample with 9.5 mm diameter air void dried at 50 °C. In comparison, crack lengths of 2.2mm and 2.3mm were measured for 0.5 w/c cement paste samples with 9.5 mm diameter air void gently dried at 55% and 75 % RH.

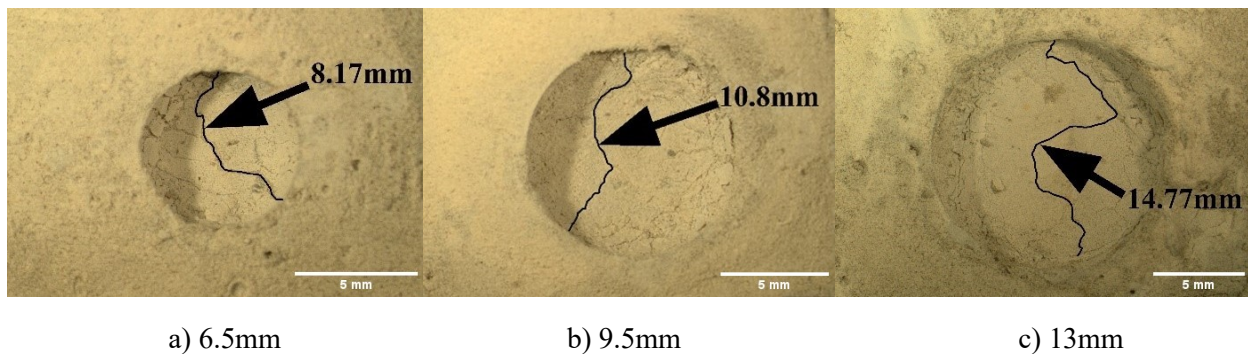
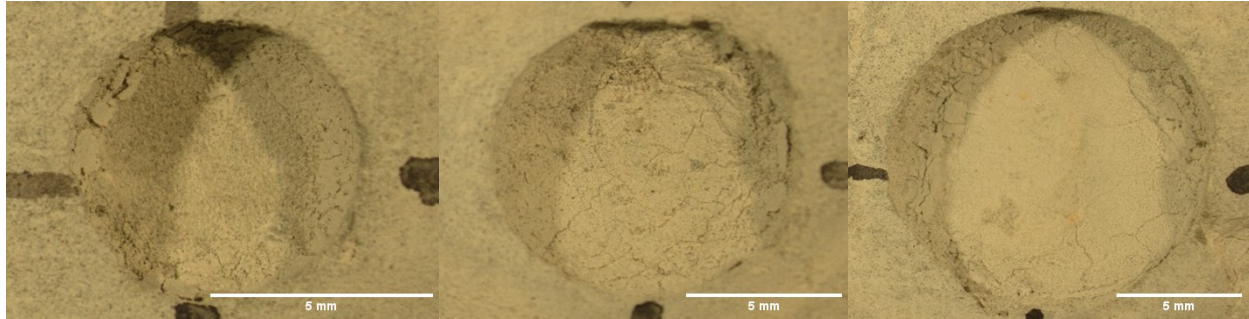


Figure 6-33 Maximum crack length formed on surfaces of air voids before conditioning at 50°C for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

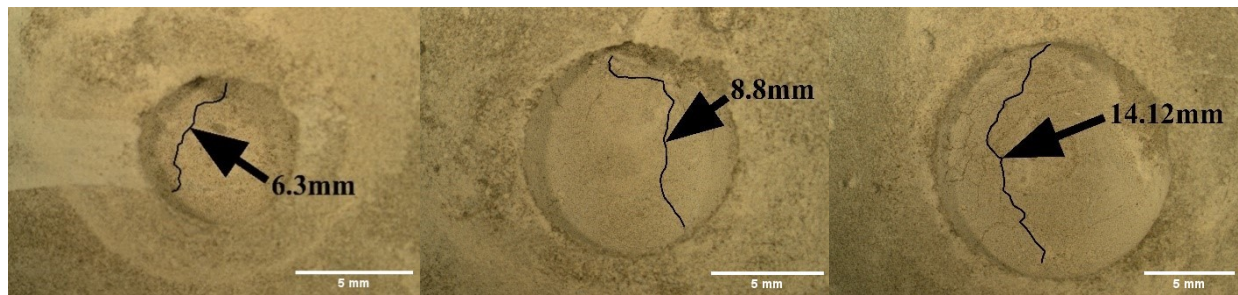


a) 6.5mm

b) 9.5mm

c) 13mm

Figure 6-34 Examples of cracks forming on surfaces of air voids after conditioning at 50°C and exposure to wetting and drying for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

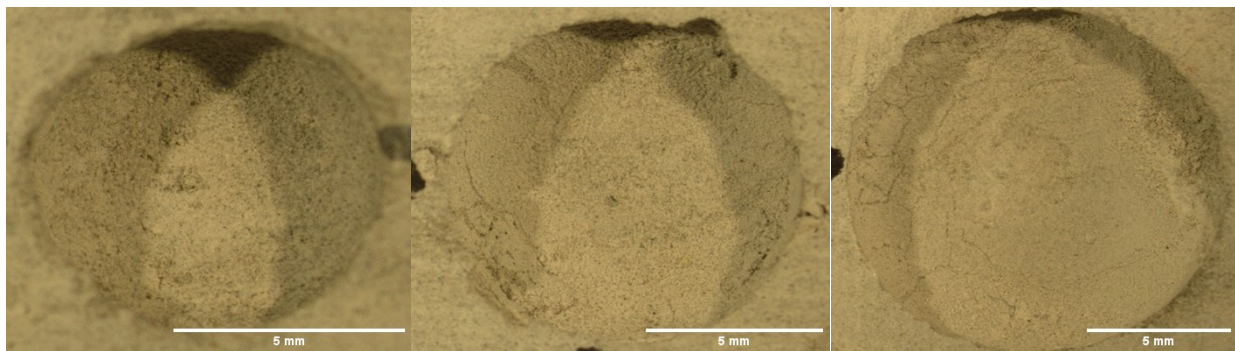


a) 6.5mm

b) 9.5mm

c) 13mm

Figure 6-35 Maximum crack length formed on surfaces of air voids before conditioning at 50°C for 0.45 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.



a) 6.5mm

b) 9.5mm

c) 13mm

Figure 6-36 Examples of cracks forming on surfaces of air voids after conditioning at 50°C and exposure to wetting and drying for 0.45 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

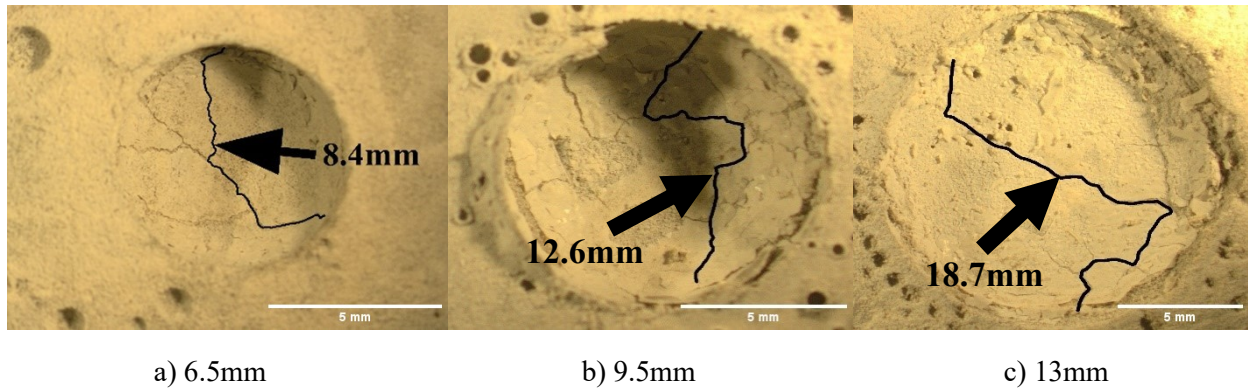


Figure 6-37 Maximum crack length formed on surfaces of air voids before gentle drying at 55% RH for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

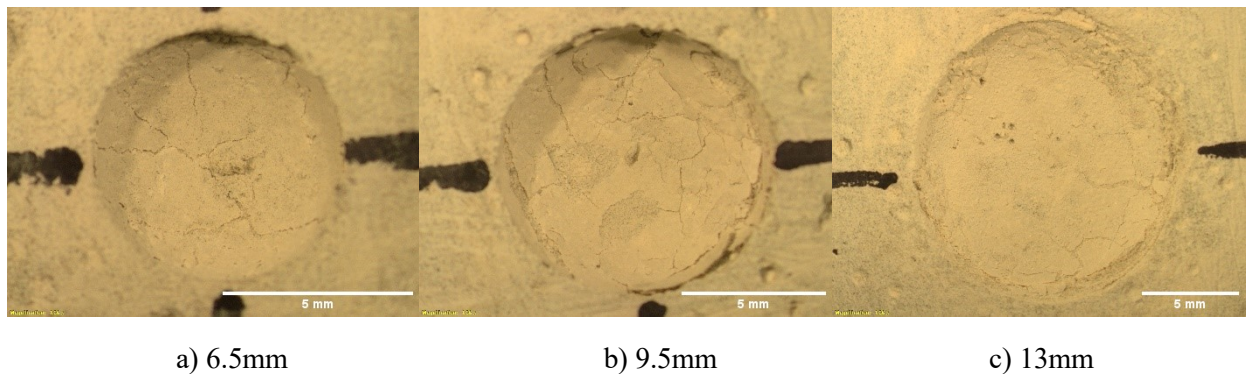


Figure 6-38 Examples of cracks forming on surfaces of air voids after gentle drying at 55% RH and exposure to wetting and drying for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

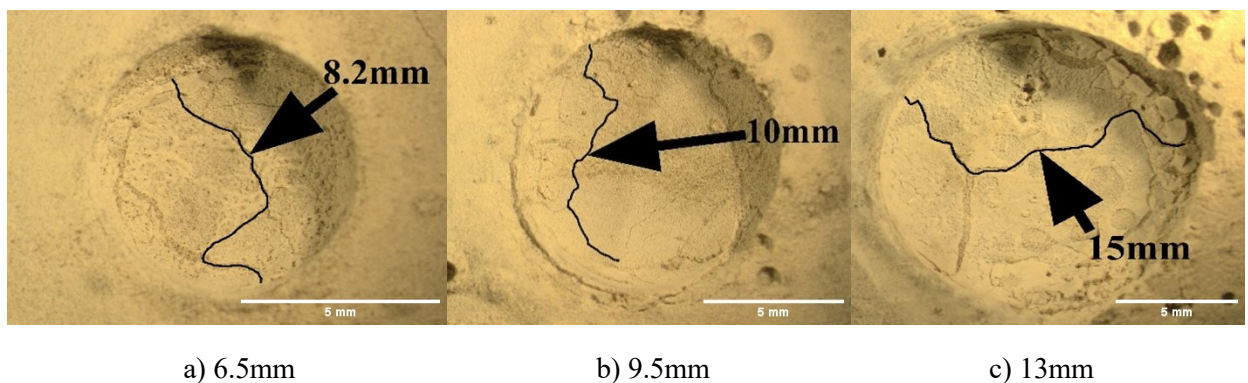
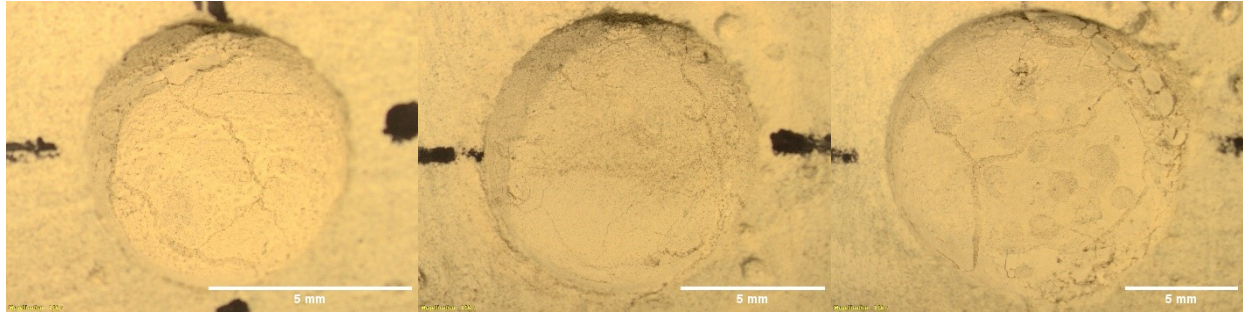


Figure 6-39 Maximum crack length formed on surfaces of air voids before gentle drying at 75% RH for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

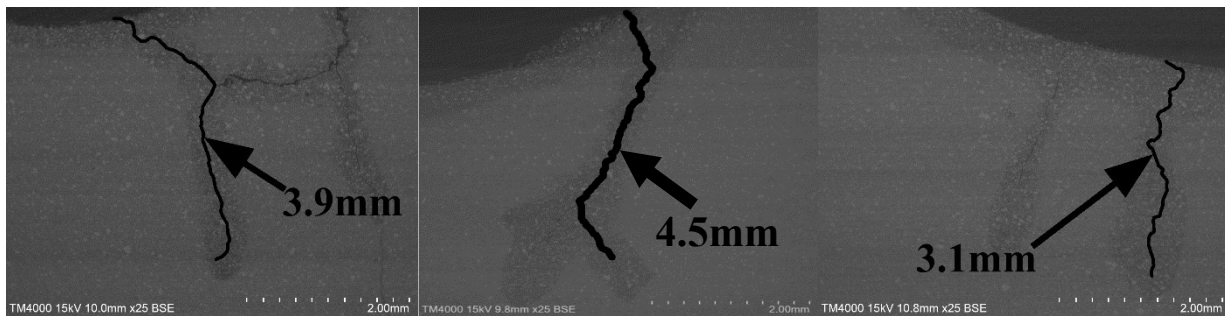


a) 6.5mm

b) 9.5mm

c) 13mm

Figure 6-40 Examples of cracks forming on surfaces of air voids after gentle drying at 55% RH and exposure to wetting and drying for 0.5 w/c cement paste specimens examined with a stereomicroscope Olympus SZX10.

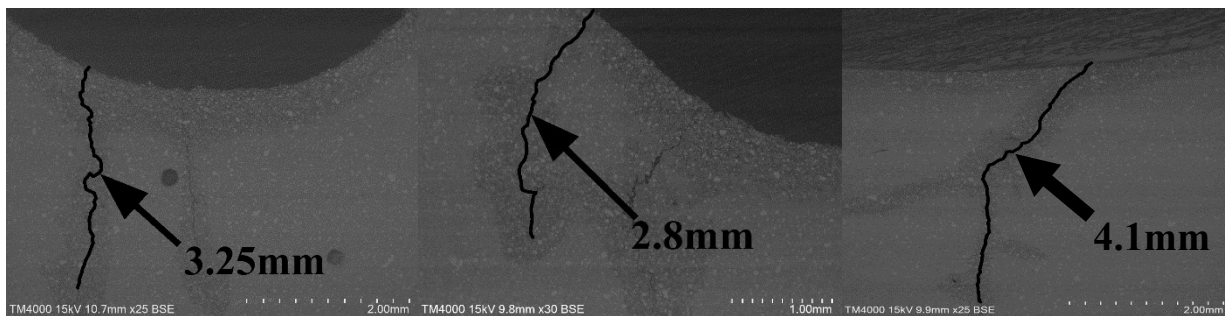


a) 6.5mm

b) 9.5mm

c) 13mm

Figure 6-41 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.5 w/c 3 days cured cement paste specimens after conditioning at 50°C and exposure to wetting and drying.



a) 6.5mm

b) 9.5mm

c) 13mm

Figure 6-42 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.45 w/c 3 days cured cement paste specimens after conditioning at 50°C and exposure to wetting and drying.

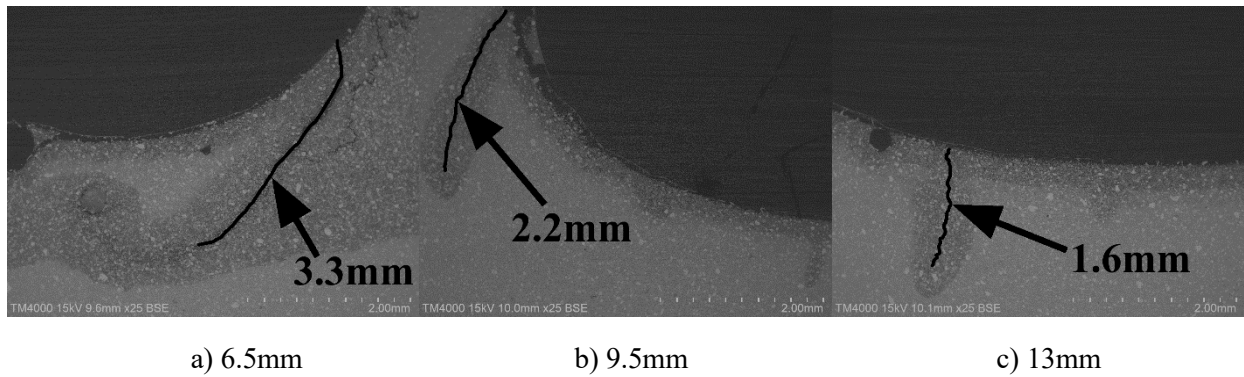


Figure 6-43 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.5 w/c 3 days cured cement paste specimens after conditioning at 55% RH and exposure to wetting and drying.

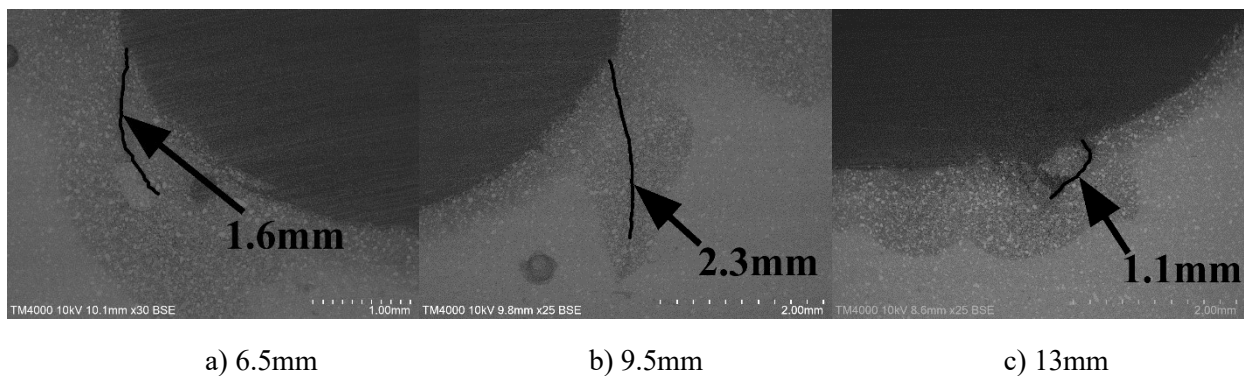


Figure 6-44 Maximum crack length formed at the paste-void interface examined with SEM/BSE for 0.5 w/c 3 days cured cement paste specimens after conditioning at 75% RH and exposure to wetting and drying.

6.6 Discussion

6.6.1 Effect of geometrical size of entrapped air voids on absorption rates

The available void space and geometrical size are critical factors that influence the time required to achieve saturation. For an identical curing age and water to cement ratio, it was observed that voids with a smaller diameter saturated at a faster rate compared to voids with a larger diameter. This occurs when the time required to transport moisture from the surface to the surrounding air is drastically shortened when distance is reduced. Similarly, numerical and experimental studies have shown an increase in the inflow

velocity of water for voids with a smaller diameter (Khanzadeh Moradillo, Hu, and Ley 2017; Wilson, Hoff, and Hall 1991).

In addition, MEMS sensors detected a greater increase in relative humidity for 13mm diameter air voids conditioned at 50°C. These results strongly suggest that the presence of microcracks can influence the diffusivity of moisture. From surface morphology images captured in Figures 6-33 and 6-41, an increase in crack length was observed for larger entrapped air voids. This is resulted by the inability to withstand high surface tension forces when cement paste hardens. Unlike smaller air bubbles that are capable of sustaining higher surface tension forces. This phenomenon has been well examined and studied with experimental studies and models based on bubble collapse dynamics to predict the collapse time frames and shapes by exerting external forces (Aganin et al. 2016; Lauterborn and Ohl 1997; Yang, Jaw, and Yeh 2009). On the other hand, it is also important to note an increased length of microcracks at higher water to cement ratios with faster rates of saturation. These findings appear to be consistent with available studies that monitored water ingress into cracks (Michel and Pease 2018; Zheng et al. 2022). For example, Michel and Pease (2018) induced cracks into concrete with a rigid wedge using a crack mouth opening displacement control (0.10 - 0.4mm). From x-ray attenuation measurements, they discovered that cracks provide a free surface for moisture ingress.

6.6.2 Comparing internal relative humidity with cumulative absorption measurements

With a known relative humidity and cumulative absorption measurement, the diffusivity of vapour into air voids can be studied. Results from this study showed a spike in relative humidity for specimens with and without voids during the initial uptake of water with an approximate rate of diffusivity of $10^{-9} \text{ m}^2/\text{s}$. These findings are in agreement with numerical and experimental studies (Bohris et al. 1998; Bordallo, Aldridge, and Desmedt 2006; Fleury et al. 2020; Strangfeld 2020). Bordallo et al. (2006) discovered that unbound water confined in pore structures will diffuse at the rate of approximately $10^{-9} \text{ m}^2/\text{s}$ and a reduction in diffusivity of $10^{-10} \text{ m}^2/\text{s}$ with the presence of gel pore water.

Additionally, findings from this study indicated that the presence of moisture in capillary pores can accelerate the rate of saturation of air voids as shown for specimens conditioned at 75% and 55 % RH. Larger suction pressure and lower viscous resistance for partially saturated capillary pore systems will transport moisture from larger to smaller pores until equilibrium is reached (Krus 1995). These findings

are similar to observations reported by various researchers (Rucker-Gramm and Beddoe 2010; S. F. Wong et al. 2001).

6.6.3 Comparing internal relative humidity with cumulative desorption measurements

During desorption, moisture will dissipate from larger pores to smaller pores. Hence, a greater reduction in internal relative humidity and an increase in cumulative desorption were expected for samples with a higher abundance of accessible pores. Sorption isotherms and ^1H NMR experiments (Baroghel-Bouny 2007; Jennings, Kumar, and Sant 2015; Muller et al. 2013) demonstrated that at 85% relative humidity capillary pores will empty followed by pores larger than 10 nm. Subsequently, gel pores within low-density (LD) C-S-H at approximately 85% to 50% relative humidity. This is reflected in the results presented in this study with a slower decrease in relative humidity for samples with greater curing age (Figure 6-31). Similarly, relative humidity decreased at a faster rate for larger entrapped air voids (Figures 6-31 and 6-32). This is also attributed to an increased crack length; thus, allowing a faster diffusivity of moisture.

6.7 Conclusions

The transport of moisture and surface morphology of air voids simulated in cementitious material were examined. Variables included w/c ratio (0.35, 0.45 and 0.5), curing age (3, 14 and 28 days), void diameter (6.5, 9.5 and 13mm) and drying conditions (50°C, 55% RH and 75% RH). The study was an attempt to explore the effects of surface cracks and geometrical sizes on moisture transport, with the overall aim to better understand whether air voids can become saturated over time.

The main findings are as follows:

a) For 50°C dried cement paste samples, vapour will preferably diffuse into smaller void spaces for air voids with diameters smaller than 9.5mm. For void diameters greater than 9.5mm, vapour diffusion was influenced by microcracks.

- b) Vapour will preferably diffuse into smaller air voids before larger air voids for cement paste samples conditioned at 55% and 75% relative humidity.
- c) During the initial uptake of water, it was evident that vapour diffuses into air voids before capillary pores become saturated. This suggests that interfacial voids can become saturated at earlier time intervals at 100 – 256 minutes.
- d) Near identical cumulative absorption and desorption measurements were observed for samples with or without surface voids.
- e) Compared to cumulative absorption and desorption measurements, RFID MEMS sensors were able to detect changes in relative humidity from induced surface cracks in air voids with a diameter of 6.5 – 13 mm. This was attributed to the high sensitivity properties and the ability to characterize moisture content within the millimeter range.

Chapter 7 Conclusion and Future Work

Limited studies have developed non-destructive techniques to monitor the moisture state at the steel-concrete interface. There is also a lack of research on understanding the effects of cementitious materials on the degradation and performance of sensor components. Therefore, the aim of this thesis was to develop a suitable and reliable non-destructive technique to monitor relative humidity at the steel-concrete interface. The developed technique can help to clarify relationships between moisture in the bulk concrete and in entrapped air voids at the surface of the steel that influence corrosion initiation and provide a more reliable assessment of corrosion risk.

This thesis confirms that RFID MEMS sensors can be embedded in enclosures at the steel-concrete interface for laboratory-based research and can be potentially applied for in-situ monitoring of real structures. The technical challenge of particulate contamination from unreacted cement and aggregate particles was overcome by introducing additive manufacturing processes and techniques. Performance of RFID MEMS sensors was unaffected after 7 months of embedment in low and high-strength concrete; however, silicon can dissolve to form Si-OH complexes after exposure to cement paste pore solution. Direct measurements of relative humidity were obtained at the steel-concrete interface and for air voids simulated in cement paste samples subjected to wetting and drying cycles. This was achieved by creating protocols and non-destructive methods to conduct systematic controlled experiments.

The main conclusions arising from this thesis are as follows:

- RFID MEMS sensors placed in incubators containing saturated sodium nitrate (70 % RH), potassium hydrogen sulphate (86 % RH), potassium nitrate (93 % RH) salt solutions and moisture (100 % RH) suggest that precise measurements can be obtained with a maximum difference of ± 2 %. However, hysteresis and inaccuracies still persist in environments above 93 % relative humidity due to condensation and stiction.

- An increase in cover depth, water to cement ratio and aggregate volume did not reduce the communication range and transmitted power of sensor technologies operating at the 13.56 MHz frequency. Relative humidity measurements at the steel-concrete interface were obtainable for low and high-strength concrete specimens with 10, 25 and 50 mm nominal cover and 8 mm diameter steel bar.
- The performance of RFID MEMS sensors was not affected after 7 months of embedment in low and high-strength concrete and 1 year of cyclic wetting and drying. However, SEM-BSE and EDS analysis indicated potassium, sodium and calcium elements present on silicon thin films and voids formed on capacitors and resistors after embedment in concrete. Environments of high alkalinity, pH 12.5 - 13, can dissolve silicon thin films to form Si-OH complexes and oxidize solder material underneath resistors to create surface voids.
- During the initial uptake of water, it was evident that vapour diffuses into air voids before capillary pores become saturated. This suggests that interfacial voids can become saturated at earlier time intervals at 100 – 256 minutes.
- Compared to cumulative absorption and desorption measurements, RFID MEMS sensors were able to detect changes in relative humidity from induced surface cracks in air voids with a diameter of 6.5 – 13 mm. This was attributed to the high sensitivity properties and the ability to characterize moisture content within the millimeter range.

RFID MEMS sensors were embedded in high and low-strength concrete with various curing ages, nominal cover depths and water to cement ratio and were found to be suitable for studying relative humidity changes at the steel-concrete interface. In the future, MEMS sensors can be integrated with chloride ion and pH sensors. The ability to monitor these parameters will provide a more informed assessment of the electrochemical behaviour during corrosion initiation and help to reduce uncertainties in durability engineering.

In addition, there are no clear indications of appropriate methods and procedures to embed sensor technologies in cementitious materials. Several issues have been identified in this thesis that needs to be addressed to improve measured results. Despite the advances of current 3D printing techniques, parts are constructed at an accuracy of the millimetre scale; thus, creating empty spaces between sensor components. This can be overcome by hydrogel infusion additive manufacturing (HIAM) by reducing the volume of empty space and distance between the sensor and steel surface with individual components printed at the microscale. This manufacturing method holds much promise but has yet to see a widescale implementation beyond small-scale prototypes of octet lattice structures of copper, nickel, silver and alloys (Tabatabai and Aljuboori 2017). The lack of mass-produced products is hindered by the multistep and time-consuming process of converting hydrogels to metal and alloys. Furthermore, microelectromechanical (MEMS) sensors can be replaced by nanoelectromechanical (NEMS) sensors to enhance the spatial resolution and distribution of local characteristics at the steel-concrete interface. Nevertheless, costly fabrication methods currently prevent this sensor technology from further development and application.

RFID MEMS sensors have demonstrated the potential for in-situ monitoring of real structures with reliable measurements obtained. This recognises that non-marine structures and spray zones in marine structures generally experience less extreme moisture gradients and less rapid changes than imposed in this study. Nonetheless, sensors are expected to be embedded for longer periods to provide appropriate monitoring of reinforced concrete structures. Hence, the reliability of sensor components needs to be further studied before this technique can be implemented confidently for in-situ monitoring. This includes an investigation into the influence of enclosure placement and the presence of rebar on distortions and accuracy of measurements. Moreover, results indicated the degradation of sensor components when exposed to cementitious materials. Thus, the application of protective coatings such as CrTiN to enhance the performance and durability of sensor components needs to be further investigated. This includes understanding the influence of protective coatings on the rate of response, precision and accuracy of measurements.

This study shows that RFID MEMS sensors were able to monitor an increase in relative humidity in air voids before samples achieve saturation. Over an extended period of time, saturated air can condense and form thin layers of water film on surfaces of voids that can influence corrosion rates. Thus, internal relative humidity measurements obtained from sensors can be complemented with neutron imaging to visualize the accumulation of moisture in macroscopic interfacial voids. Furthermore, to enhance the

durability performance of modern structures CEM I is frequently partially replaced by supplementary cementitious materials (SCMs), e.g., fly ash, ground granulated blast furnace slag and silica fume. SCMs may improve particle packing and generate additional hydration products that refine and densify the overall microstructure. Therefore, it is reasonable to expect a lower moisture content in macroscopic interfacial voids which still requires more research to evaluate such properties.

References

- Abd El Haleem, S M, S Abd El Wanees, E E Abd El Aal, and A Diab. 2010. "Environmental Factors Affecting the Corrosion Behavior of Reinforcing Steel II. Role of Some Anions in the Initiation and Inhibition of Pitting Corrosion of Steel in Ca(OH)₂ Solutions." *Corrosion Science* 52(2): 292–302. <https://www.sciencedirect.com/science/article/pii/S0010938X09004375>.
- Aganin, A A, M A Ilgamov, L A Kosolapova, and V G Malakhov. 2016. "Dynamics of a Cavitation Bubble near a Solid Wall." *Thermophys. Aeromech. (Germany)* 23(2): 211–20. <http://dx.doi.org/10.1134/S0869864316020074>.
- Alam, Mohammed A, Michael H Azarian, and Michael G Pecht. 2012. "Embedded Capacitors in Printed Wiring Board: A Technological Review." *Journal of Electronic Materials* 41(8): 2286–2303. <https://doi.org/10.1007/s11664-012-2044-3>.
- Angst, Ueli. 2011. "Chloride Induced Reinforcement Corrosion in Concrete: Concept of Critical Chloride Content – Methods and Mechanisms." Norwegian University of Science and Technology. <http://hdl.handle.net/11250/236720>.
- Angst, Ueli M. et al. 2017. "The Steel–Concrete Interface." *Materials and Structures/Materiaux et Constructions* 50(2): 143 (24 pp.).
- . 2019. "The Effect of the Steel–Concrete Interface on Chloride-Induced Corrosion Initiation in Concrete: A Critical Review by RILEM TC 262-SCI." *Materials and Structures* 52(88).
- Angst, Ueli M et al. 2022. "Beyond the Chloride Threshold Concept for Predicting Corrosion of Steel in Concrete." *Applied Physics Reviews* 9: 11321. <https://doi.org/10.1063/5.0076320>.
- Angst, Ueli M, Bernhard Elsener, Claus K Larsen, and Oystein Vennesland. 2011. "Chloride Induced Reinforcement Corrosion: Electrochemical Monitoring of Initiation Stage and Chloride Threshold Values." *Corrosion Science* 53(4): 1451–64. <http://dx.doi.org/10.1016/j.corsci.2011.01.025>.
- Asp, A et al. 2019. "Impact of Different Concrete Types on Radio Propagation: Fundamentals and Practical RF Measurements." In *2019 4th International Conference on Smart and Sustainable Technologies (SpliTech)*, , 1–8.
- Barber, Peter et al. 2009. "Polymer Composite and Nanocomposite Dielectric Materials for Pulse Power

- Energy Storage.” *Materials* 2(4): 1697–1733. <https://www.mdpi.com/1996-1944/2/4/1697>.
- Baroghel-Bouny, Veronique. 2007. “Water Vapour Sorption Experiments on Hardened Cementitious Materials. Part I: Essential Tool for Analysis of Hygral Behaviour and Its Relation to Pore Structure.” *Cement and Concrete Research* 37(3): 414–37.
<http://dx.doi.org/10.1016/j.cemconres.2006.11.019>.
- Bilgutay, N, J Popovics, S Popovics, and M Karaoguz. 2001. “Recent Developments in Concrete Nondestructive Evaluation.” In *2001 IEEE International Conference on Acoustics, Speech, and Signal Processing. Proceedings (Cat. No.01CH37221)*, , 3393–96 vol.6.
- Birleanu, C et al. 2017. “Relative Humidity Effect on Pull-off Forces in MEMS Flexible Structures Measured by AFM.” In *2017 Symposium on Design, Test, Integration and Packaging of MEMS/MOEMS (DTIP)*, Piscataway, NJ, USA, 8 pp. <http://dx.doi.org/10.1109/DTIP.2017.7984478>.
- Bjorninen, Toni, Jukka Voutilainen, Lauri Sydanheimo, and Leena Ukkonen. 2013. “Characterization of HF RFID Tags Exposed to Moisture Based on Threshold Power Measurement.” In *Progress in Electromagnetics Research Symposium*, Progress in Electromagnetics Research Symposium, Department of Electronics and Communications Engineering, Tampere University of Technology, P. O. Box 692, FI-33101, Tampere, FinlandVoyantic Ltd., Kutojantie 11, FI-02630, Espoo, Finland: Electromagnetics Academy, 815–18.
- Bohris, A J et al. 1998. “A Broad Line NMR and MRI Study of Water and Water Transport in Portland Cement Pastes.” *Magnetic Resonance Imaging* 16(5): 455–61.
<https://www.sciencedirect.com/science/article/pii/S0730725X98000721>.
- Bolton, Joshua et al. 2017. “Performance and Benchmarking of Multisurface UHF RFID Tags for Readability and Reliability” ed. Aldo Minardo. *Journal of Sensors*: 3467593.
<https://doi.org/10.1155/2017/3467593>.
- Bordallo, Heloisa N, Laurence P Aldridge, and Arnaud Desmedt. 2006. “Water Dynamics in Hardened Ordinary Portland Cement Paste or Concrete: From Quasielastic Neutron Scattering.” *The Journal of Physical Chemistry B* 110(36): 17966–76. <https://doi.org/10.1021/jp062922f>.
- Bouzaffour, K et al. 2021. “Development of an Embedded UHF-RFID Corrosion Sensor for Monitoring Corrosion of Steel in Concrete.” *IEEE Sensors Journal* 21(10): 12306–12.
- Brazzle, J D et al. 2003. “A Hysteresis-Free Platinum Alloy Flexure Material for Improved Performance and Reliability of MEMS Devices.” In *TRANSDUCERS '03. 12th International Conference on*

Solid-State Sensors, Actuators and Microsystems. Digest of Technical Papers (Cat. No.03TH8664), , 1152–55.

- Broas, M et al. 2015. “Galvanic Corrosion of Silicon-Based Thin Films: A Case Study of a MEMS Microphone.” In *2015 IEEE 65th Electronic Components and Technology Conference (ECTC). Proceedings*, Piscataway, NJ, USA, 453–59. <http://dx.doi.org/10.1109/ECTC.2015.7159630>.
- Broomfield, John p. 2007. *Corrosion of Steel In Concrete*. 2nd ed. Taylor & Francis.
- Brownjohn, J M W, and P Moyo. 2001. “Monitoring of Singapore-Malaysia Second Link during Construction.” In *Proceedings of the SPIE - The International Society for Optical Engineering*, Proc. SPIE - Int. Soc. Opt. Eng. (USA), Nanyang Technol. Univ., Sch. of Civil & Struct. Eng., Singapore BT - Second International Conference on Experimental Mechanics, 29 Nov.-1 Dec. 2000: SPIE-Int. Soc. Opt. Eng., 528–33. <http://dx.doi.org/10.1117/12.429633>.
- Bullard, Jeffrey W et al. 2011. “Mechanisms of Cement Hydration.” *Cement and Concrete Research* 41(12): 1208–23. <https://www.sciencedirect.com/science/article/pii/S0008884610002152>.
- C128-15, ASTM. 2016. “ASTM C128-15 Standard Test Method for Relative Density (Specific Gravity) and Absorption of Fine Aggregate.”
- C566-19, ASTM. 2019. “ASTM C566-19 Standard Test Method for Total Evaporable Moisture Content of Aggregate by Drying.”
- Cano-Barrita, P.F. de J. et al. 2009. “Embedded NMR Sensors to Monitor Evaporable Water Loss Caused by Hydration and Drying in Portland Cement Mortar.” *Cement and Concrete Research* 39(4): 324–28. <http://dx.doi.org/10.1016/j.cemconres.2009.01.011>.
- Cano-Barrita, P F de J et al. 2004. “Moisture Distribution in Drying Ordinary and High Performance Concrete Cured in a Simulated Hot Dry Climate.” *Materials and Structures* 37(8): 522–31. <https://doi.org/10.1007/BF02481576>.
- Cano-Barrita, Prisciliano F, and Floriberto Díaz-Díaz. 2021. “Moisture Distribution during Water Absorption of Ordinary Portland Cement Mortars Obtained with Low-Field Unilateral Magnetic Resonance.” *Materials* 14(15).
- Chai, Jing, Qi Liu, Jinxuan Liu, and Dingding Zhang. 2018. “Optical Fiber Sensors Based on Novel Polyimide for Humidity Monitoring of Building Materials.” *Optical Fiber Technology* 41: 40–47. <http://dx.doi.org/10.1016/j.yofte.2017.12.013>.

- Chen, Yu-Min, and Mark E Orazem. 2016. "Impedance Analysis of ASTM A416 Tendon Steel Corrosion in Alkaline Simulated Pore Solutions." *Corrosion Science* 104: 26–35.
<https://www.sciencedirect.com/science/article/pii/S0010938X15301694>.
- Choudhary, Nitin, D K Kharat, and Davinder Kaur. 2011. "Surface Modification of NiTi/PZT Heterostructure Thin Films Using Various Protective Layers for Potential MEMS Applications." *Surface and Coatings Technology* 206(7): 1735–43.
<http://dx.doi.org/10.1016/j.surfcoat.2011.08.056>.
- Crassous, Jérôme, Matteo Ciccotti, and Elisabeth Charlaix. 2011. "Capillary Force between Wetted Nanometric Contacts and Its Application to Atomic Force Microscopy." *Langmuir* 27(7): 3468–73.
<https://doi.org/10.1021/la103781y>.
- Dai, Ching-Liang. 2007. "A Capacitive Humidity Sensor Integrated with Micro Heater and Ring Oscillator Circuit Fabricated by CMOS–MEMS Technique." *Sensors and Actuators B: Chemical* 122(2): 375–80. <https://www.sciencedirect.com/science/article/pii/S0925400506004369>.
- . 2007. "A Nanowire WO₃ Humidity Sensor Integrated with Micro-Heater and Inverting Amplifier Circuit on Chip Manufactured Using CMOS-MEMS Technique." *Sensors and Actuators B: Chemical* 123(2): 896–901.
<https://www.sciencedirect.com/science/article/pii/S0925400506007337>.
- Dalton, Laura E, Jacob M LaManna, Scott Jones, and Mohammad Pour-Ghaz. 2022. "Does ITZ Influence Moisture Transport in Concrete?" *Transport in Porous Media* 144(3): 623–39.
<https://doi.org/10.1007/s11242-022-01826-z>.
- Daly, D, T Melia, and G Baldwin. 2010. "Concrete Embedded RFID for Way-Point Positioning." In *2010 International Conference on Indoor Positioning and Indoor Navigation*, , 1–10.
- Dauti, Dorjan et al. 2018. "Analysis of Moisture Migration in Concrete at High Temperature through In-Situ Neutron Tomography." *Cement and Concrete Research* 111: 41–55.
<https://www.sciencedirect.com/science/article/pii/S0008884618301613>.
- Deschner, Florian et al. 2012. "Hydration of Portland Cement with High Replacement by Siliceous Fly Ash." *Cement and Concrete Research* 42(10): 1389–1400.
<http://dx.doi.org/10.1016/j.cemconres.2012.06.009>.
- Donahoe, Daniel N, Michael Pecht, Isabel K Lloyd, and Sanka Ganesan. 2006. "Moisture Induced Degradation of Multilayer Ceramic Capacitors." *Microelectronics Reliability* 46(2): 400–408.

<https://www.sciencedirect.com/science/article/pii/S0026271405001022>.

- Donno, Danilo De, Luca Catarinucci, Adolfo Di Serio, and and Luciano Tarricone. 2013. "A Long-Range Computational RFID Tag for Temperature and Acceleration Sensing Applications." *Progress in Electromagnetics Research C* 45: 223–35.
- Egerton, R F, P Li, and M Malac. 2004. "Radiation Damage in the TEM and SEM." *Micron* 35(6): 399–409. <https://www.sciencedirect.com/science/article/pii/S0968432804000381>.
- Erdogan, S T, X Nie, P E Stutzman, and E J Garboczi. 2010. "Micrometer-Scale 3-D Shape Characterization of Eight Cements: Particle Shape and Cement Chemistry, and the Effect of Particle Shape on Laser Diffraction Particle Size Measurement." *Cement and Concrete Research* 40(5): 731–39. <http://dx.doi.org/10.1016/j.cemconres.2009.12.006>.
- Eun, Ye-Jin et al. 2018. "Archaeal Cells Share Common Size Control with Bacteria despite Noisier Growth and Division." *Nature Microbiology* 3(2): 148–54. <https://doi.org/10.1038/s41564-017-0082-6>.
- Fagerlund, Göran. 1993. *The Long Time Water Absorption in the Air-Pore Structure of Concrete*.
- Feldkamp, L A, L C Davis, and J W Kress. 1984. "Practical Cone-Beam Algorithm." *J. Opt. Soc. Am. A* 1(6): 612–19. <https://opg.optica.org/josaa/abstract.cfm?URI=josaa-1-6-612>.
- Fischer, N, R Haerdtl, and P J McDonald. 2015. "Observation of the Redistribution of Nanoscale Water Filled Porosity in Cement Based Materials during Wetting." *Cement and Concrete Research* 68: 148–55. <http://dx.doi.org/10.1016/j.cemconres.2014.10.013>.
- Fleury, M et al. 2020. "Water Diffusion Measurements in Cement Paste, Mortar and Concrete Using a Fast NMR Based Technique." *Construction and Building Materials* 259. <http://dx.doi.org/10.1016/j.conbuildmat.2020.119843>.
- Gajewicz, Agata M. 2014. "Characterisation of Cement Microstructure and Pore - Water Interaction by H Nuclear Magnetic Resonance Relaxometry." University of Surrey.
- Ghods, P et al. 2011. "XPS Depth Profiling Study on the Passive Oxide Film of Carbon Steel in Saturated Calcium Hydroxide Solution and the Effect of Chloride on the Film Properties." *Applied Surface Science* 257(10): 4669–77. <https://www.sciencedirect.com/science/article/pii/S0169433210018829>.
- Ghods, P, O B Isgor, G A McRae, and G P Gu. 2010. "Electrochemical Investigation of Chloride-Induced Depassivation of Black Steel Rebar under Simulated Service Conditions." *Corrosion Science* 52(5):

- 1649–59. <https://www.sciencedirect.com/science/article/pii/S0010938X10000752>.
- Glass, G K, B Reddy, and N R Buenfeld. 2000. “The Participation of Bound Chloride in Passive Film Breakdown on Steel in Concrete.” *Corrosion Science* 42(11): 2013–21.
[http://dx.doi.org/10.1016/S0010-938X\(00\)00040-8](http://dx.doi.org/10.1016/S0010-938X(00)00040-8).
- Goldstein, Joseph I. et al. 2007. *Scanning Electron Microscopy and X-Ray Microanalysis*. 3rd ed. Springer New York, NY.
- Gu, Lei, Qing-An Huang, and Ming Qin. 2004. “A Novel Capacitive-Type Humidity Sensor Using CMOS Fabrication Technology.” *Sensors and Actuators B: Chemical* 99(2–3): 491–98.
<https://www.sciencedirect.com/science/article/pii/S0925400503009584>.
- Guo, F L, X Niu, and B B He. 2015. “An Analytical Study on Steam-Driven Delamination and Stability of Delamination Growth in Electronic Packages.” *Engineering Fracture Mechanics* 144: 89–100.
<https://www.sciencedirect.com/science/article/pii/S0013794415003343>.
- Hon, Melitta et al. 2008. “Cathodic Corrosion of Polycrystalline Silicon MEMS.” *Sensors and Actuators A: Physical* 145–146: 323–29.
<https://www.sciencedirect.com/science/article/pii/S0924424707008989>.
- Hong, Shuxian et al. 2020. “Visualized Tracing of Capillary Absorption Process in Cementitious Material Based on X Ray Computed Tomography.” *CEMENT & CONCRETE COMPOSITES* 107.
- Horne, A. T., I. G. Richardson, and R. M.D. Brydson. 2007a. “Quantitative Analysis of the Microstructure of Interfaces in Steel Reinforced Concrete.” *Cement and Concrete Research* 37(12): 1613–23.
- Horne, A T, I G Richardson, and R M D Brydson. 2007b. “Quantitative Analysis of the Microstructure of Interfaces in Steel Reinforced Concrete.” *Cement and Concrete Research* 37(12): 1613–23.
<https://www.sciencedirect.com/science/article/pii/S0008884607001950>.
- Hu, Yan et al. 2017. “Absolute Three-Dimensional Micro Surface Profile Measurement Based on a Greenough-Type Stereomicroscope.” *Measurement Science and Technology* 28(4): 45004.
<https://dx.doi.org/10.1088/1361-6501/aa5a2d>.
- ISO/IEC 15693-2:2019 Cards and Security Devices for Personal Identification — Contactless Vicinity Objects — Part 2: Air Interface and Initialization*. 2019. International Organization for Standardization.

- J., McCarter W, Watson D W., and Chrisp T M. 2001. "Surface Zone Concrete: Drying, Absorption, and Moisture Distribution." *Journal of Materials in Civil Engineering* 13(1): 49–57.
[https://doi.org/10.1061/\(ASCE\)0899-1561\(2001\)13:1\(49\)](https://doi.org/10.1061/(ASCE)0899-1561(2001)13:1(49)).
- J I Izpura. 2001. "Side Contact Effects on the Capacitance Properties of Junction Devices. Application to III-Nitrogen Structures." *Semiconductor Science and Technology* 16(4): 243.
<https://dx.doi.org/10.1088/0268-1242/16/4/310>.
- Jennings, Hamlin M, Aditya Kumar, and Gaurav Sant. 2015. "Quantitative Discrimination of the Nano-Pore-Structure of Cement Paste during Drying: New Insights from Water Sorption Isotherms." *Cement and Concrete Research* 76: 27–36. <http://dx.doi.org/10.1016/j.cemconres.2015.05.006>.
- Johnson, Sarah J, and F Rohan Walker. 2015. "Strategies to Improve Quantitative Assessment of Immunohistochemical and Immunofluorescent Labelling." *Scientific Reports* 5(1): 10607.
<https://doi.org/10.1038/srep10607>.
- Jorapur, Nikhil et al. 2015. "A Low-Power, Low-Cost Soil-Moisture Sensor Using Dual-Probe Heat-Pulse Technique." *Sensors and Actuators A: Physical* 233: 108–17.
<https://www.sciencedirect.com/science/article/pii/S0924424715300479>.
- Joubert, O et al. 1991. "Resist Degradation under Plasma Exposure: Synergistic Effects of Ion Bombardment." *J. Appl. Phys. (USA)* 69(3): 1697–1702. <http://dx.doi.org/10.1063/1.347214>.
- Kahn, H, C Deeb, I Chasiotis, and A H Heuer. 2005. "Anodic Oxidation during MEMS Processing of Silicon and Polysilicon: Native Oxides Can Be Thicker than You Think." *Journal of Microelectromechanical Systems* 14(5): 914–23.
- Käthler, Carolina Boschmann, Ueli M. Angst, and Bernhard Elsener. 2018. "Towards Understanding Corrosion Initiation in Concrete-Influence of Local Concrete Properties in the Steel-Concrete Interfacial Zone." In *MATEC Web of Conferences*, , 04002 (6 pp.).
- Khanzadeh Moradllo, M, Qiang Hu, and M T Ley. 2017. "Using X-Ray Imaging to Investigate in-Situ Ion Diffusion in Cementitious Materials." *Constr. Build. Mater. (Netherlands)* 136: 88–98.
<http://dx.doi.org/10.1016/j.conbuildmat.2017.01.038>.
- Kong, Qingzhao et al. 2013. "Very Early Age Concrete Hydration Characterization Monitoring Using Piezoceramic Based Smart Aggregates." *Smart Materials and Structures* 22(8): 85025.
<https://dx.doi.org/10.1088/0964-1726/22/8/085025>.
- Krumm, M, S Kasperl, and M Franz. 2008. "Reducing Non-Linear Artifacts of Multi-Material Objects in

- Industrial 3D Computed Tomography.” *NDT & E International* 41(4): 242–51.
<https://www.sciencedirect.com/science/article/pii/S0963869507001478>.
- Krus, Martin. 1995. “Moisture Transport and Storage Coefficients of Porous Mineral Building Materials, Theoretical Principles and New Test Methods.” University of Stuttgart.
- Kwiecińska, Barbara, Sławomira Pusz, and Brett J Valentine. 2019. “Application of Electron Microscopy TEM and SEM for Analysis of Coals, Organic-Rich Shales and Carbonaceous Matter.” *International Journal of Coal Geology* 211: 103203.
<https://www.sciencedirect.com/science/article/pii/S0166516218311571>.
- Lal, Sreeyuth et al. 2014. “Investigation of Water Uptake in Porous Asphalt Concrete Using Neutron Radiography.” *Transport in Porous Media* 105(2): 431–50. <https://doi.org/10.1007/s11242-014-0376-6>.
- Lauterborn, Werner, and Claus-Dieter Ohl. 1997. “Cavitation Bubble Dynamics.” *Ultrasonics Sonochemistry* 4(2): 65–75. <https://www.sciencedirect.com/science/article/pii/S1350417797000096>.
- Leemann, Andreas, Peter Nygaard, Josef Kaufmann, and Roman Loser. 2015. “Relation between Carbonation Resistance, Mix Design and Exposure of Mortar and Concrete.” *Cement and Concrete Composites* 62: 33–43. <https://www.sciencedirect.com/science/article/pii/S0958946515000967>.
- Li, Kefei, Chunqiu Li, and Zhaoyuan Chen. 2009. “Influential Depth of Moisture Transport in Concrete Subject to Drying–Wetting Cycles.” *Cement and Concrete Composites* 31(10): 693–98.
<https://www.sciencedirect.com/science/article/pii/S0958946509001413>.
- Li, Yunjia et al. 2013. “Large Stroke Staggered Vertical Comb-Drive Actuator for the Application of a Millimeter-Wave Tunable Phase Shifter.” *Journal of Microelectromechanical Systems* 22(4): 962–75. <http://dx.doi.org/10.1109/JMEMS.2013.2252146>.
- . 2018. “Challenges and Issues of Using Polymers as Structural Materials in MEMS: A Review.” *Journal of Microelectromechanical Systems* 27(4): 581–98.
<http://dx.doi.org/10.1109/JMEMS.2018.2837684>.
- Liu, Tiejun et al. 2013. “Exploratory Study on Water Seepage Monitoring of Concrete Structures Using Piezoceramic Based Smart Aggregates.” *Smart Materials and Structures* 22(6): 65002.
<https://dx.doi.org/10.1088/0964-1726/22/6/065002>.
- Livesu, Marco et al. 2017. “From 3D Models to 3D Prints: An Overview of the Processing Pipeline.” *Computer Graphics Forum* 36(2): 537–64. <https://doi.org/10.1111/cgf.13147>.

- Lu, Hong M, Chuck Goldsmith, Lawrence Cauller, and Jeong-Bong Lee. 2007. "MEMS-Based Inductively Coupled RFID Transponder for Implantable Wireless Sensor Applications." *IEEE Transactions on Magnetics* 43(6): 2412–14.
- Lu, Xin, Konstantin Hicke, Mathias Breithaupt, and Christoph Strangfeld. 2021. "Distributed Humidity Sensing in Concrete Based on Polymer Optical Fiber." *Polymers* 13(21). <http://dx.doi.org/10.3390/polym13213755>.
- Malan, F. S., K. Ahmet, A. Dunstert, and R. Hearing. 2004. "Development of a Microwave Frequency Sensor for the Long-Term Localised Moisture Monitoring of Concrete." *Magazine of Concrete Research* 56(5): 263–71.
- Marcotte, Tracy Dawn. 2001. "Characterization of Chloride-Induced Corrosion Products That Form in Steel-Reinforced Cementitious Materials." University of Waterloo.
- Mazzanti, Valentina, Lorenzo Malagutti, and Francesco Mollica. 2019. "FDM 3D Printing of Polymers Containing Natural Fillers: A Review of Their Mechanical Properties." *Polymers* 11(7).
- McCarter, W. J. et al. 2012. "Developments in Performance Monitoring of Concrete Exposed to Extreme Environments." *Journal of Infrastructure Systems* 18(3): 167–75.
- Michel, A, and B J Pease. 2018. "Moisture Ingress in Cracked Cementitious Materials." *Cem. Concr. Res. (Netherlands)* 113: 154–68. <http://dx.doi.org/10.1016/j.cemconres.2018.08.009>.
- Mikolajek, Morten et al. 2019. "Fabrication and Characterization of Fully Inkjet Printed Capacitors Based on Ceramic/Polymer Composite Dielectrics on Flexible Substrates." *Scientific Reports* 9(1): 13324. <https://doi.org/10.1038/s41598-019-49639-3>.
- Miller, David C et al. 2006. "Galvanic Corrosion: A Microsystems Device Integrity and Reliability Concern." In *Proc.SPIE*, , 611105. <https://doi.org/10.1117/12.644932>.
- Miyake, Norio et al. 2019. "Development Overview and Practical Applications in Stereomicroscope with 'Perfect Zoom System.'" In *Proc.SPIE*, , 1110608. <https://doi.org/10.1117/12.2530547>.
- Mohammed, Tarek Uddin, Nobuaki Otsuki, Hidenori Hamada, and Toru Yamaji. 2002. "Chloride-Induced Corrosion of Steel Bars in Concrete with Presence of Gap at Steel-Concrete Interface." *ACI Materials Journal* 99(2): 149–56.
- Morettini, Giulia, Massimiliano Palmieri, Lorenzo Capponi, and Luca Landi. 2022. "Comprehensive Characterization of Mechanical and Physical Properties of PLA Structures Printed by FFF-3D-

- Printing Process in Different Directions.” *Progress in Additive Manufacturing* 7(5): 1111–22.
<https://doi.org/10.1007/s40964-022-00285-8>.
- Muller, A C A et al. 2015. “Influence of Silica Fume on the Microstructure of Cement Pastes: New Insights from ¹H NMR Relaxometry.” *Cement and Concrete Research* 74: 116–25.
<http://dx.doi.org/10.1016/j.cemconres.2015.04.005>.
- Muller, A C A, K L Scrivener, A M Gajewicz, and P J McDonald. 2013. “Use of Bench-Top NMR to Measure the Density, Composition and Desorption Isotherm of C-S-H in Cement Paste.” *Microporous and Mesoporous Materials* 178: 99–103.
<http://dx.doi.org/10.1016/j.micromeso.2013.01.032>.
- Muqaibel, A, A Safaai-Jazi, A Bayram, and S M Riad. 2003. “Ultra Wideband Material Characterization for Indoor Propagation.” In *IEEE Antennas and Propagation Society International Symposium. Digest. Held in Conjunction with: USNC/CNC/URSI North American Radio Sci. Meeting (Cat. No.03CH37450)*, , 623–26 vol.4.
- Nahar, R K. 2000. “Study of the Performance Degradation of Thin Film Aluminum Oxide Sensor at High Humidity.” *Sensors and Actuators B: Chemical* 63(1): 49–54.
<https://www.sciencedirect.com/science/article/pii/S0925400599005110>.
- Nazeri, Muhammad Firdaus Mohd, and Ahmad Azmin Mohamad. 2015. “Effect of Exposure to Alkaline Solution on Sn–9Zn Solder Joints.” *Journal of Materials Processing Technology* 219: 164–72.
<https://www.sciencedirect.com/science/article/pii/S0924013614005147>.
- Nestle, Nikolaus, Petrik Galvosas, and Jorg Karger. 2007. “Liquid-Phase Self-Diffusion in Hydrating Cement Pastes - Results from NMR Studies and Perspectives for Further Research.” *Cement and Concrete Research* 37(3): 398–413. <http://dx.doi.org/10.1016/j.cemconres.2006.02.004>.
- Nguyen, A Q, G Klysz, F Deby, and J.-P. Balayssac. 2017. “Evaluation of Water Content Gradient Using a New Configuration of Linear Array Four-Point Probe for Electrical Resistivity Measurement.” *Cement and Concrete Composites* 83: 308–22.
<http://dx.doi.org/10.1016/j.cemconcomp.2017.07.020>.
- Noh, Bo-In, Jong-Bum Lee, and Seung-Boo Jung. 2008. “Effect of Surface Finish Material on Printed Circuit Board for Electrochemical Migration.” *Microelectronics Reliability* 48(4): 652–56.
<https://www.sciencedirect.com/science/article/pii/S0026271407004283>.
- Noh, Bo-In, Jeong-Won Yoon, Won-Sik Hong, and Seung-Boo Jung. 2009. “Evaluation of

- Electrochemical Migration on Flexible Printed Circuit Boards with Different Surface Finishes.” *Journal of Electronic Materials* 38(6): 902–7. <https://doi.org/10.1007/s11664-009-0737-z>.
- Nokken, Michelle R, and R Douglas Hooton. 2002. “Dependence of Rate of Absorption on Degree of Saturation of Concrete.” *Cement, Concrete and Aggregates* 24(1): 20–24.
- Norris, Ashley, Mohamed Saafi, and Peter Romine. 2008. “Temperature and Moisture Monitoring in Concrete Structures Using Embedded Nanotechnology/Microelectromechanical Systems (MEMS) Sensors.” *Construction and Building Materials* 22(2): 111–20.
- Oesch, Tyler, Frank Weise, Dietmar Meinel, and Christian Gollwitzer. 2019. “Quantitative In-Situ Analysis of Water Transport in Concrete Completed Using X-Ray Computed Tomography.” *Transport in Porous Media* 127(2): 371–89. <https://doi.org/10.1007/s11242-018-1197-9>.
- Padala, Suresh Kumar, Shashank Bishnoi, and Bishwajit Bhattacharjee. 2022. “Long-Term Moisture Penetration in Concrete Exposed to Marine Tidal Conditions.” *Magazine of Concrete Research* 74(14): 740–55. <https://doi.org/10.1680/jmacr.21.00188>.
- Patton, S T, W D Cowan, and J S Zabinski. 1999. “Performance and Reliability of a New MEMS Electrostatic Lateral Output Motor.” In *37th Annual IEEE International Reliability Physics Symposium*, 1999 IEEE International Reliability Physics Symposium Proceedings. 37th Annual (Cat. No.99CH36296), US Air Force Res. Lab., Wright-Patterson AFB, OH, United States BT - 1999 IEEE International Reliability Physics Symposium Proceedings. 37th Annual, 23-25 March 1999: IEEE, 179–88. <http://dx.doi.org/10.1109/RELPHY.1999.761610>.
- Pedersen, C et al. 2004. “Highly Reliable O-Ring Packaging Concept for MEMS Pressure Sensors.” *Sensors and Actuators A: Physical* 115(2–3): 617–27. <https://www.sciencedirect.com/science/article/pii/S092442470400281X>.
- Polder, R B. 2001. “Test Methods for on Site Measurement of Resistivity of Concrete- A RILEM TC-154 Technical Recommendation.” *Construction and Building Materials* 15(2–3): 125–31.
- Poursae, A, and C M Hansson. 2007. “Reinforcing Steel Passivation in Mortar and Pore Solution.” *Cement and Concrete Research* 37(7): 1127–33. <https://www.sciencedirect.com/science/article/pii/S0008884607000993>.
- Prakash, Guru, Ayan Sadhu, Sriram Narasimhan, and Jean-Marie Brehe. 2018. “Initial Service Life Data towards Structural Health Monitoring of a Concrete Arch Dam.” *Structural Control and Health Monitoring* 25(1): e2036. <https://doi.org/10.1002/stc.2036>.

- Raman, A, S Sahasrabudhe, L Dietz, and M Hossain. 2011. "Phenomenological Understanding of Sn Migration in Ceramic-Chip Capacitors." In *2011 IEEE 61st Electronic Components and Technology Conference (ECTC)*, , 541–46.
- Raunima, Tuomas, Eero Tuominen, and Juha Vinha. 2019. "Behavior of Capacitive Humidity Sensors in Monitoring the Drying of Concrete Walls." In *MATEC Web of Conferences*, , 02053.
- Reimer, Ludwig. 2013. *Scanning Electron Microscopy - Physics of Image Formation and Microanalysis*. 2nd ed. Springer Berlin, Heidelberg.
- Rezayat, H et al. 2015. "Structure–Mechanical Property Relationship in Fused Deposition Modelling." *Materials Science and Technology* 31(8): 895–903.
<https://doi.org/10.1179/1743284715Y.0000000010>.
- Rossi, A, G Puddu, and B Elsener. 2006. "The Surface of Iron and Fe10Cr Alloys in Alkaline Media." In *Corrosion of Reinforcement in Concrete: Monitoring, Prevention and Rehabilitation Techniques*, Corrosion of Reinforcement in Concrete: Monitoring, Prevention and Rehabilitation Techniques, Department of Inorganic and Analytical Chemistry, University of Cagliari, I-09042 Monserrato, Cagliari, Sardinia, Italy: Elsevier Ltd, 44–61. <http://dx.doi.org/10.1533/9781845692285.44>.
- Rossi, E et al. 2020. "The Influence of Defects at the Steel/Concrete Interface for Chloride-Induced Pitting Corrosion of Naturally-Deteriorated 20-Years-Old Specimens Studied through X-Ray Computed Tomography." *Construction and Building Materials* 235: 481–91.
<http://dx.doi.org/10.1016/j.conbuildmat.2019.117474>.
- Rucker-Gramm, P, and R E Beddoe. 2010. "Effect of Moisture Content of Concrete on Water Uptake." *Cement and Concrete Research* 40(1): 102–8.
<https://www.sciencedirect.com/science/article/pii/S0008884609002415>.
- Saafi, Mohamed, and Peter Romine. 2005. "Preliminary Evaluation of MEMS Devices for Early Age Concrete Property Monitoring." *Cement and Concrete Research* 35(11): 2158–64.
- Sakonkanapong, Arnon, and Chuwong Phongcharoenpanich. 2020. "Near-Field HF-RFID and CMA-Based Circularly Polarized Far-Field UHF-RFID Integrated Tag Antenna" ed. María Elena de Cos Gómez. *International Journal of Antennas and Propagation* 2020: 6427157.
<https://doi.org/10.1155/2020/6427157>.
- Samaha, Hani R, and Kenneth C Hover. 1992. "Influence of Microcracking on the Mass Transport Properties of Concrete." *ACI Materials Journal* 89(4): 416–24.

- Sánchez, M et al. 2007. “Electrochemical Impedance Spectroscopy for Studying Passive Layers on Steel Rebars Immersed in Alkaline Solutions Simulating Concrete Pores.” *Electrochimica Acta* 52(27): 7634–41. <https://www.sciencedirect.com/science/article/pii/S0013468607002162>.
- Sandberg, P. 1998. *The Effect of Defects at the Steel-Concrete Interface, Exposure Regime and Cement Type on Pitting Corrosion in Concrete*. <http://lup.lub.lu.se/record/1529238/file/1895826.pdf>.
- Schindelin, Johannes et al. 2012. “Fiji: An Open-Source Platform for Biological-Image Analysis.” *Nature Methods* 9(7): 676–82. <https://doi.org/10.1038/nmeth.2019>.
- Seo, Wonil, Yong-Ho Ko, Young-Ho Kim, and Sehoon Yoo. 2019. “Void Fraction of a Sn–Ag–Cu Solder Joint underneath a Chip Resistor and Its Effect on Joint Strength and Thermomechanical Reliability.” *Journal of Materials Science: Materials in Electronics* 30(17): 15889–96. <https://doi.org/10.1007/s10854-019-01935-4>.
- Singh, Madhusudan, Hanna M Haverinen, Parul Dhagat, and Ghassan E Jabbour. 2010. “Inkjet Printing—Process and Its Applications.” *Advanced Materials* 22(6): 673–85. <https://onlinelibrary.wiley.com/doi/abs/10.1002/adma.200901141>.
- Smith, Scott H., Matthieu Vandamme, and Kimberly E. Kurtis. 2020. “Dissolution Kinetics of Trapped Air in a Spherical Void: Modeling the Long-Term Saturation of Cementitious Materials.” *Cement and Concrete Research* 130(February): 105996. <https://doi.org/10.1016/j.cemconres.2020.105996>.
- Sokoll, Thorsten, and Arne F. Jacob. 2014. “Self-Oscillating Measurement System for in Situ-Monitoring Moisture in Buildings.” *IEEE Transactions on Instrumentation and Measurement* 63(12): 2911–18.
- Song, Xiaobing, Qiming Kong, and Xila Liu. 2007. “Experimental Study on Chloride Threshold Levels in OPC.” *Tumu Gongcheng Xuebao/China Civil Engineering Journal* 40(11): 59–63.
- Standard, British. 2001. “BS 7973-1: Spacers and Chairs for Steel Reinforcement and Their Specification.” *BSI Standards Publication*.
- Starosvetsky, D, M Kovler, and Y Ein-Eli. 2004. “Perspective on Si Negative Potential Dissolution Mechanism.” *Electrochemical and Solid-State Letters* 7(8): G168. <https://dx.doi.org/10.1149/1.1766252>.
- Strangfeld, Christoph. 2020. “Determination of the Diffusion Coefficient and the Hydraulic Conductivity of Porous Media Based on Embedded Humidity Sensors.” *Construction and Building Materials* 263. <http://dx.doi.org/10.1016/j.conbuildmat.2020.120092>.

- Strangfeld, Christoph, Sergej Johann, and Matthias Bartholmai. 2019. "Smart RFID Sensors Embedded in Building Structures for Early Damage Detection and Long-Term Monitoring." *Sensors (Switzerland)* 19(24): 5514 (18 pp.).
- Sun, Q, G M Rizvi, C T Bellehumeur, and P Gu. 2008. "Effect of Processing Conditions on the Bonding Quality of FDM Polymer Filaments." *Rapid Prototyping Journal* 14(2): 72–80.
<https://doi.org/10.1108/13552540810862028>.
- Surendar, A et al. 2018. "Effect of Solder Layer Thickness on Thermo-Mechanical Reliability of a Power Electronic System." *Journal of Materials Science: Materials in Electronics* 29: 15249–58.
<https://doi.org/10.1007/s10854-018-9667-y>.
- T E Everhart, and R F M Thornley. 1960. "Wide-Band Detector for Micro-Microampere Low-Energy Electron Currents." *Journal of Scientific Instruments* 37(7): 246. <https://dx.doi.org/10.1088/0950-7671/37/7/307>.
- Tabatabai, Habib, and Mohammed Aljuboori. 2017. "A Novel Concrete-Based Sensor for Detection of Ice and Water on Roads and Bridges." *Sensors (Switzerland)* 17(12).
- Tan, Kara-Anne et al. 2016. "Choroidal Vascularity Index – a Novel Optical Coherence Tomography Parameter for Disease Monitoring in Diabetes Mellitus?" *Acta Ophthalmologica* 94(7): e612–16.
<https://doi.org/10.1111/aos.13044>.
- Tanner, Danelle et al. 2000. *MEMS Reliability: Infrastructure, Test Structures, Experiments, and Failure Modes*. United States. <https://www.osti.gov/biblio/750344>.
- Tanner, Danelle M et al. 2000. "MEMS Reliability in Shock Environments." In *Annual Proceedings - Reliability Physics (Symposium)*, Sandia Natl Lab, Albuquerque, United States: IEEE, 129–38.
- Taufik, Mohammad, and Prashant K Jain. 2022. "Part Surface Quality Improvement Studies in Fused Deposition Modelling Process: A Review." *Australian Journal of Mechanical Engineering* 20(2): 527–51. <https://doi.org/10.1080/14484846.2020.1723342>.
- Toropovs, N et al. 2015. "Real-Time Measurements of Temperature, Pressure and Moisture Profiles in High-Performance Concrete Exposed to High Temperatures during Neutron Radiography Imaging." *Cement and Concrete Research* 68: 166–73.
<https://www.sciencedirect.com/science/article/pii/S0008884614002233>.
- Trtik, P et al. 2011. "Release of Internal Curing Water from Lightweight Aggregates in Cement Paste Investigated by Neutron and X-Ray Tomography." *Nuclear Instruments and Methods in Physics*

- Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment* 651(1): 244–49. <https://www.sciencedirect.com/science/article/pii/S0168900211002907>.
- Villagrán Zaccardi, Y. A., J. Fulla García, P. Huélamo, and Á A. Di Maio. 2009. “Influence of Temperature and Humidity on Portland Cement Mortar Resistivity Monitored with Inner Sensors.” *Materials and Corrosion* 60(4): 294–99.
- Volpi, Enrico, Andrea Olietti, Matteo Stefanoni, and Stefano P Trasatti. 2015. “Electrochemical Characterization of Mild Steel in Alkaline Solutions Simulating Concrete Environment.” *Journal of Electroanalytical Chemistry* 736: 38–46. <https://www.sciencedirect.com/science/article/pii/S157266571400469X>.
- Wang, Biyun, Pamela Faure, Mickael Thiery, and Veronique Baroghel-Bouny. 2013. “¹H NMR Relaxometry as an Indicator of Setting and Water Depletion during Cement Hydration.” *Cement and Concrete Research* 45(1): 1–14. <http://dx.doi.org/10.1016/j.cemconres.2012.10.017>.
- Wierig, H J. 1984. “Longtime Studies on the Carbonation on Concrete under Normal Outdoor Exposure.” In *PROCEEDINGS OF SEMINAR DURABILITY OF CONCRETE STRUCTURES UNDER NORMAL OUTDOOR EXPOSURE*, , 239–49.
- Williamson, Jon, and O Burkan Isgor. 2016. “The Effect of Simulated Concrete Pore Solution Composition and Chlorides on the Electronic Properties of Passive Films on Carbon Steel Rebar.” *Corrosion Science* 106: 82–95. <https://www.sciencedirect.com/science/article/pii/S0010938X16300270>.
- Wilson, Moira A, W D Hoff, and Christopher Hall. 1991. “Water Movement in Porous Building Materials. X. Absorption from a Small Cylindrical Cavity.” *Building and Environment* 26(2): 143–52. [http://dx.doi.org/10.1016/0360-1323\(91\)90021-3](http://dx.doi.org/10.1016/0360-1323(91)90021-3).
- Wittmann, F. 1968. “Surface Tension Shrinkage and Strength of Hardened Cement Paste.” *Materiaux et Constructions, Materials and Structures* 1(6): 547–52.
- Wong, H S et al. 2010. “On the Penetration of Corrosion Products from Reinforcing Steel into Concrete Due to Chloride-Induced Corrosion.” *Corrosion Science* 52(7): 2469–80. <http://dx.doi.org/10.1016/j.corsci.2010.03.025>.
- Wong, H S, and N R Buenfeld. 2006. “Patch Microstructure in Cement-Based Materials: Fact or Artefact?” *Cement and Concrete Research* 36(5): 990–97. <https://www.sciencedirect.com/science/article/pii/S0008884606000317>.

- Wong, Hong S. et al. 2022. “Methods for Characterising the Steel–Concrete Interface to Enhance Understanding of Reinforcement Corrosion: A Critical Review by RILEM TC 262-SCI.” *Materials and Structures* 55(124).
- Wong, Hong Seong. 2006. “Quantifying the Pore Structure of Cement-Based Materials Using Backscattered Electron Microscopy.” Imperial College London.
- Wong, S F, T H Wee, S Swaddiwudhipong, and S L Lee. 2001. “Study of Water Movement in Concrete.” *Magazine of Concrete Research* 53(3): 205–20. <https://doi.org/10.1680/macrc.2001.53.3.205>.
- Wu, Z., H. S. Wong, C. Chen, and N. R. Buenfeld. 2019. “Anomalous Water Absorption in Cement-Based Materials Caused by Drying Shrinkage Induced Microcracks.” *Cement and Concrete Research* 115(June 2018): 90–104.
- Yamaji, Y, K Sugano, O Tabata, and T Tsuchiya. 2007. “Tensile-Mode Fatigue Tests and Fatigue Life Predictions of Single Crystal Silicon in Humidity Controlled Environments.” In *2007 20th IEEE International Conference on Micro Electro Mechanical Systems - MEMS '07*, Piscataway, NJ, USA, 267–70.
- Yang, Sheng-Hsueh, Shenq-Yuh Jaw, and Keh-Chia Yeh. 2009. “Single Cavitation Bubble Generation and Observation of the Bubble Collapse Flow Induced by a Pressure Wave.” *Experiments in Fluids* 47(2): 343–55. <https://doi.org/10.1007/s00348-009-0670-1>.
- Yeo, T. L., D. Eckstein, et al. 2006. “Demonstration of a Fibre-Optic Sensing Technique for the Measurement of Moisture Absorption in Concrete.” *Smart Materials and Structures* 15(2).
- Yeo, T. L., M. A.C. Cox, et al. 2006. “Optical Fiber Sensors for Monitoring Ingress of Moisture in Structural Concrete.” *Review of Scientific Instruments* 77(5).
- Yio, M H N, H S Wong, and N R Buenfeld. 2016. “3D Monte Carlo Simulation of Backscattered Electron Signal Variation across Pore-Solid Boundaries in Cement-Based Materials.” *Cement and Concrete Research* 89: 320–31. <https://www.sciencedirect.com/science/article/pii/S0008884616305117>.
- You, Zhanping, Julian Mills-Beale, Brandon D. Pereles, and Keat Ghee Ong. 2008. “A Wireless, Passive Embedded Sensor for Real-Time Monitoring of Water Content in Civil Engineering Materials.” *IEEE Sensors Journal* 8(12): 2053–58.
- Zaghoul, U et al. 2011. “Different Stiction Mechanisms in Electrostatic MEMS Devices: Nanoscale Characterization Based on Adhesion and Friction Measurements.” In *2011 16th International Solid-State Sensors, Actuators and Microsystems Conference*, , 2478–81.

- Zemmour, H et al. 2015. “Impact of Soil on UWB Buried Antenna and Communication Link in IR-UWB WUSN Applications.” In *2015 European Radar Conference (EuRAD)*, , 353–56.
- Zemmour, H, G Baudoin, and A Diet. 2016. “Effect of Depth and Soil Moisture on Buried Ultra-Wideband Antenna.” *Electronics Letters* 52(10): 792–94. <https://doi.org/10.1049/el.2015.3521>.
- . 2017. “Soil Effects on the Underground-to-Aboveground Communication Link in Ultrawideband Wireless Underground Sensor Networks.” *IEEE Antennas Wirel. Propag. Lett. (USA)* 16: 218–21. <http://dx.doi.org/10.1109/LAWP.2016.2570298>.
- Zhang, Hao, Junjie Li, and Fei Kang. 2022. “Real-Time Monitoring of Humidity inside Concrete Structures Utilizing Embedded Smart Aggregates.” *Construction and Building Materials* 331: 127317. <https://www.sciencedirect.com/science/article/pii/S0950061822009977>.
- Zhang, Mingzhong. 2017. “Pore-Scale Modelling of Relative Permeability of Cementitious Materials Using X-Ray Computed Microtomography Images.” *Cement and Concrete Research* 95: 18–29. <https://www.sciencedirect.com/science/article/pii/S0008884616305853>.
- Zhang, P, F H Wittmann, T Zhao, and E H Lehmann. 2010. “Neutron Imaging of Water Penetration into Cracked Steel Reinforced Concrete.” *Physica B: Condensed Matter* 405(7): 1866–71. <https://www.sciencedirect.com/science/article/pii/S0921452610000852>.
- Zhao, Haitao et al. 2021. “Investigation of Moisture Transport in Cement-Based Materials Using Low-Field Nuclear Magnetic Resonance Imaging.” *Magazine of Concrete Research* 73(5): 252–70. <https://doi.org/10.1680/jmacr.19.00211>.
- Zheng, Fan et al. 2022. “Rapid Visualization and Quantification of Water Penetration into Cement Paste through Cracks with X-Ray Imaging.” *Cement and Concrete Composites* 125. <http://dx.doi.org/10.1016/j.cemconcomp.2021.104293>.
- Zhou, Shuangxi et al. 2016. “A Novel Passive Wireless Sensor for Concrete Humidity Monitoring.” *Sensors (Switzerland)* 16(9): 1535 (15pp.).