University of Southern Queensland
Faculty of Health, Engineering & Sciences

Effects of Elevated Temperature and Simulated Environmental Conditions on the Properties of Epoxy Based Polymer Resin

A Dissertation submitted by

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Abstract

Concrete is the most widely used material in structural engineering. However, when exposed to Australias climate, concrete can suffer stress, shrinkage cracking and deterioration. These harsh environmental conditions include excessive heat, moisture, alkalinity, and high humidity (hygrothermal environment). Thus, there is a need to determine and investigate new materials that has the potential to replace concrete in severe environmental conditions.

Epoxy is a commercially available polymer that has historically been used for crack repair and coating due to its known high strength and durability properties. However, epoxy-based polymer concrete is uneconomical and inconvenient for large civil infrastructures. The use of fillers practically reduces the price of epoxy based polymer and has been found to improve the mechanical properties. However, little research has been done to understand the effect of filers on the temperature sensitivity and durability of epoxy based polymers. This project analyses the effect of light-weight particulate filler has on the thermomechanical and durability properties of epoxy resin, with respect to simulated environmental conditions.

To achieve the research objectives, the project was divided into two studies. Study 1 was conducted to determine the optimal filler content, with respect to elevated temperature, that had no significant reduction in the compressive strength of the epoxy resin. Compression testing from room temperature to 80°C and physical observations were conducted. Study 2 aimed at evaluating the six-month durability of the optimal mix selected from Stage 1 at different simulated environmental conditions. The samples were exposed to either air, saltwater, water or hygrothermal environment. Changes in the compressive strength, appearance, dimensions, weight and microscopic structure were observed at pre-set intervals.

From Study 1 it was concluded that the optimal mix design was determined to be the 60:40. There was an overall decrease of 90.2% and 96.4% in peak stress and Youngs modulus respectively. Minimal voids were present on the specimens and compressive failure was an evenly distributed shear failure. While the 40:60 specimens had an overall decrease of 85.2% and 92.1% in peak stress and Youngs modulus respectively, the mixture can create issues in actual application due its low workability, voids and brittle compressive failure.

From Study 2 it was concluded that the optimal mix design meets six month durability exposed to simulated environmental conditions. Dimensions remained consistent and there was no more than 0.3% water absorption. With the increase of duration all specimens had an increase in peak stress, with air having a 38% increase.

For the specimens exposed to the water and salt-water environments the same trends were presented with the increase of duration. Therefore the salt had no effect on the durability of the specimens. For the specimens exposed to the hygrothermal environment, the elevated temperature resulted in post curing. After 7 days exposure, the peak stress had an increase of 20%, which was 12% higher than the specimens exposed to the other environments. Therefore the filler had no adverse effects on the durability of the specimens.

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Limitations of Use

Certification of Dissertation

I certify that the ideas, designs and experimental work, results, analyses and conclusions set out in this dissertation are entirely my own effort, except where otherwise indicated and acknowledged.
I further certify that the work is original and has not been previously submitted for assessment in any other course or institution, except where specifically stated.

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Ashlii Timms

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Chapter 1

Introduction

1.1 Background

In structural engineering concrete is the single most widely used material in the world. However, when exposed to Australias climate, concrete can suffer stress, shrinkage cracking and deterioration. These harsh environmental conditions include excessive heat, moisture, alkalinity, and high humidity (hygrothermal environment).

Epoxy is a commercially available polymer that has historically been used for crack repair and coating due to its known high strength and durability properties. This project analyses the effect of light-weight particulate filler has on the thermomechanical and durability properties of epoxy resin, with respect to simulated environmental conditions.

Despite the commercial use of epoxy for its mechanical properties (Lokuge & Aravinthan 2013), there is a decrease in those properties with increased temperature past the glass transition range (Michels et al. 2015). Ideally, the epoxy is post-cured to increase the glass transition temperature (Custdio, Broughton & Cruz 2011). However, this is uneconomical and inconvenient for large concrete structures.

The use of fillers practically reduces the price of epoxy based polymer and has been found to improve the mechanical properties. However, little research has been done to understand the effects of fillers on epoxy that has been cured at ambient temperatures. The effect of the fillers on the durability of the epoxy based polymers has also not been fully researched.

This lead to the main motivation of this study, to analyse the effect light-weight particulate filler has on the thermomechanical and durability properties of epoxy resin cured at ambient temperatures.

1.2 Scope and Limitations

In this project the epoxy specimens will consists of different percentages of epoxy resin and light-weight particulate filler. Due to concrete commonly been used in compression the specimens will be tested in compression. To simulate the Australian climate the simulated environments included in this report are air, water, salt-water and hygrothermal. To ensure that the results are economical and feasible the materials are limited to those that are commercially available. Due to the post-curing being impractical in certain circumstance the specimens will also be cured at ambient temperatures, instead of elevated.

1.3 Research Objectives

The objectives of this research are:

1. Determine the optimal filler content, with respect to elevated temperature, that had no significant reduction in compressive strength of the epoxy resin.
2. Evaluate the six-month durability of the optimal mix design with respect to simulated environmental conditions.

These objective are summarised into the following statement of the aim:

To analyse the effect light-weight particulate filler has on the thermomechanical and durability properties of epoxy resin.

1.4 Overview

The following is a brief overview of each chapter in the dissertation.

Chapter 2 Literature Review

This chapter is a review of the current literature that has been published on epoxy and the exposure environment.

Chapter 3 Methodology

This chapter provides a description of how the specimens were constructed and the testing methods used to answer the objectives of this report.

Chapter 4 Effects of Elevated Temperature on compressive behaviour of Epoxy-Based Polymer Resin

This chapter presents the results of Study 1 which seeks to answer the first objective. A discussion and analysis of the results are provided along with a summary of key findings.

Chapter 5 Effects of Simulated Environment on Durability

This chapter presents the results of Study 2 which seeks to answer the second objective. A discussion and analysis of the results are provided along with a summary of key findings.

Chapter 6 Conclusion and Recommendations

This chapter summarises the results of the project and provides recommendations, improvements and possible future works.

Chapter 2

Literature Review

2.1 Introduction

This chapter analyses literature needed to determine the effect of elevated temperature and simulated environmental conditions on the properties of PFR for structural applications. Current literature is work published regarding the Australian climate, epoxy resin and light-weight particulate filler. After completing the literature review, the following chapter focuses on the experimentation and methodologies to be used based on the implication of the research.

2.2 Australian Climate

Exposure to the Australian climate can result in concrete structures being subjected to stress, shrinkage cracking and deterioration. These harsh environmental conditions include excessive heat, moisture, alkalinity, and high humidity (hygrothermal environment). Epoxy is a commercially available polymer that has historically been used for crack repair and coating of concrete.

To answer the second objective on the six-month durability of the epoxy, service conditions of the epoxy need to be determined. Determining these service condition is required in order to experimentally simulate the environmental conditions.

2.2.1 Temperature

When determining what service temperatures the epoxy may be exposed to, no information could be found. Due to variations the service temperature would have to be determined for a case by case basis. From the Bureau of Meteorology (2015) the highest maximum temperature in the past year in Australia in illustrated in Figure 2.1.

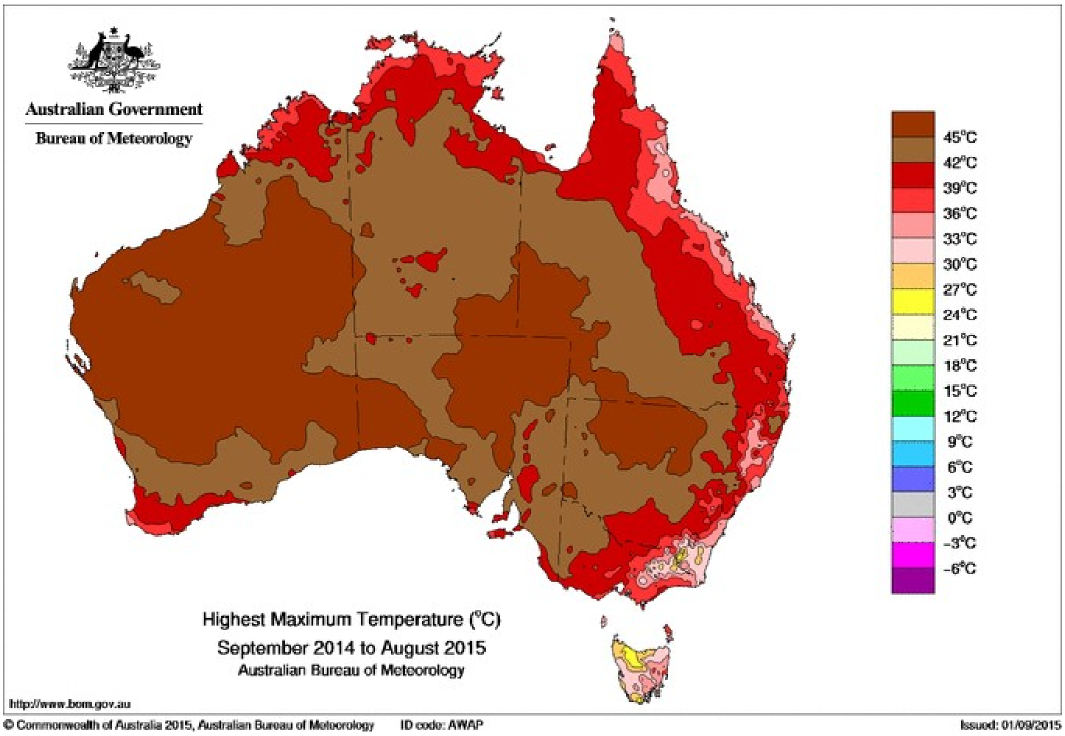

Figure 2.1: Australia's Highest Maximum Temperature in the Past Year (Bureau of Meteorology 2015)

From Figure 2.1 the maximum temperature did not exceed 50°C. This temperature, however, is only an indication of the air temperature, no the temperature the epoxy may rise to.

2.2.2 Moisture and Humidity

To determine the exposure to moisture and humidity Figures 2.2 and 2.3 illustrate the average annual rainfall and relative humidity respectively.

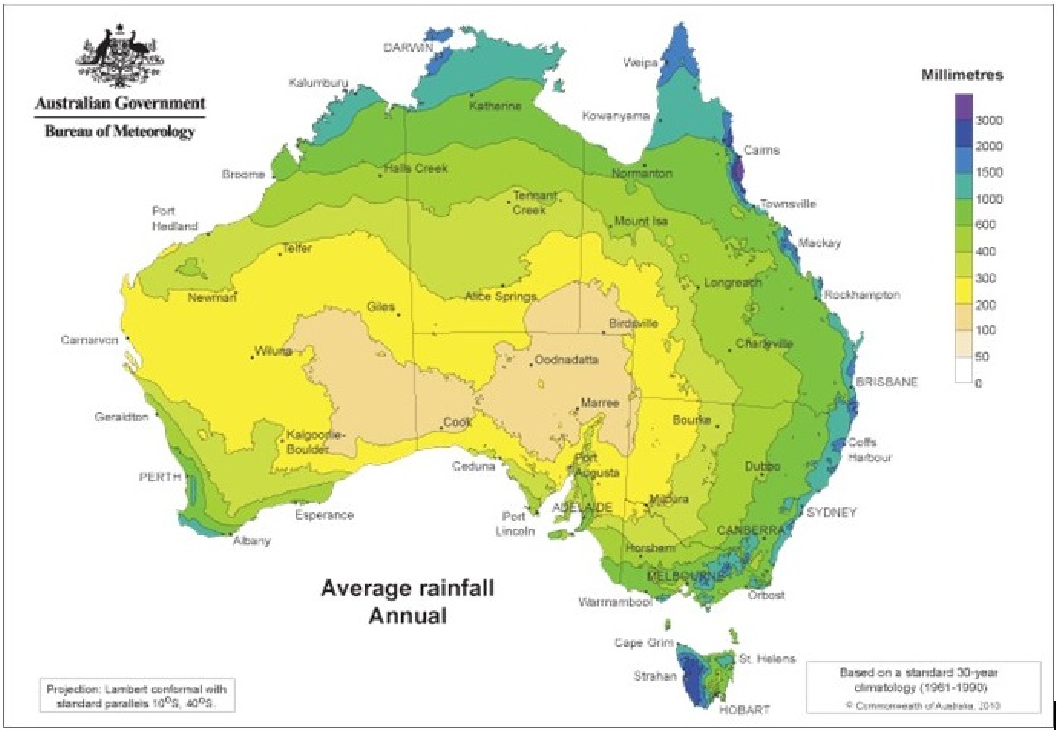

Figure 2.2: Average Annual Rainfall (Bureau of Meteorology 2015)

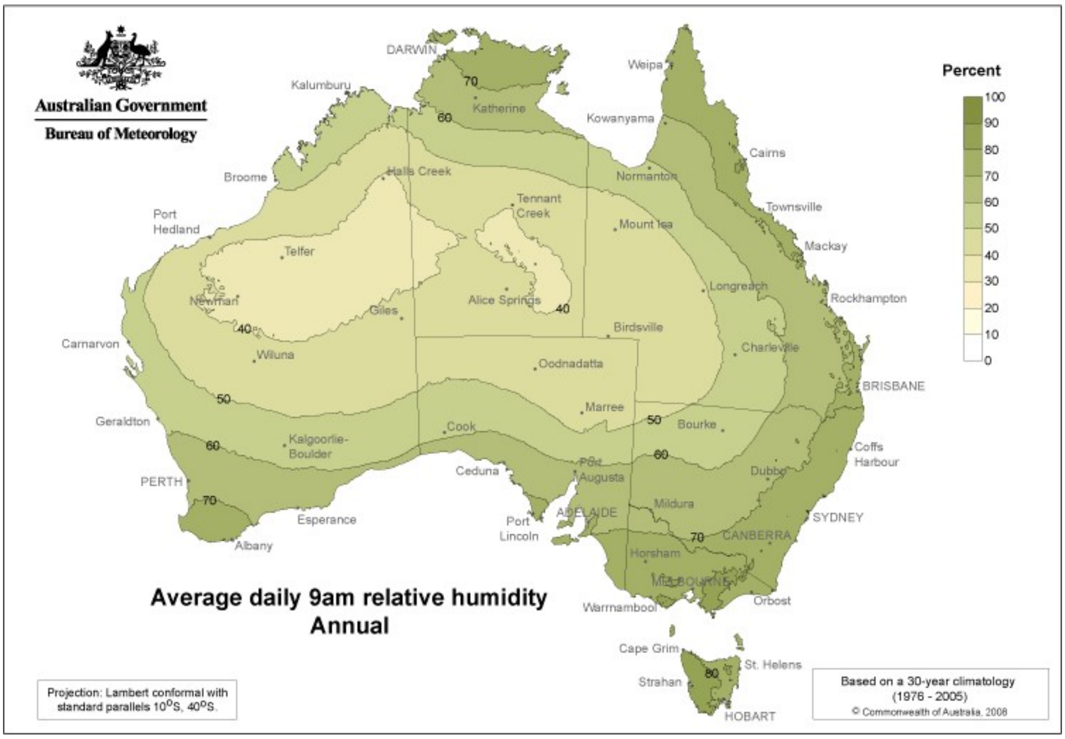

Figure 2.3: Average Annual Relative Humidity (Bureau of Meteorology 2015)

In Figure 2.3 the relative humidity is an indicator of the moisture content of the air. Figure 2.3 illustrate the amount of moisture the air can contain as a percentage (Bureau of Meteorology 2015). According to Reis & Ferreira (2005) tropical climates are the most aggressive due to the combined temperature and humidity. This suggest a hygrothermal environment which is the combination of temperature and water, which can simulate a muggy environment. According to Karalekas, Cugnoni & Botsis (2009), exposure to a hygrothermal environment is require to determine moisture absorption at higher temperatures. Moisture absorption can negatively effect thermomechanical properties and consequently durability and reliability.

Figures 2.2 and 2.3 are only an indication of possible exposure conditions. Moisture exposure could be due to rainfall or constant exposure in dams for example. In it assumed that the water would have impurities in it, therefore, tap water could be used to simulate a constant moisture exposure.

2.2.3 Alkalinity

In coastal regions the epoxy would be exposed to sea-water, which is an alkaline environment. Figure 2.4 illustrates the global salinity of sea-water.

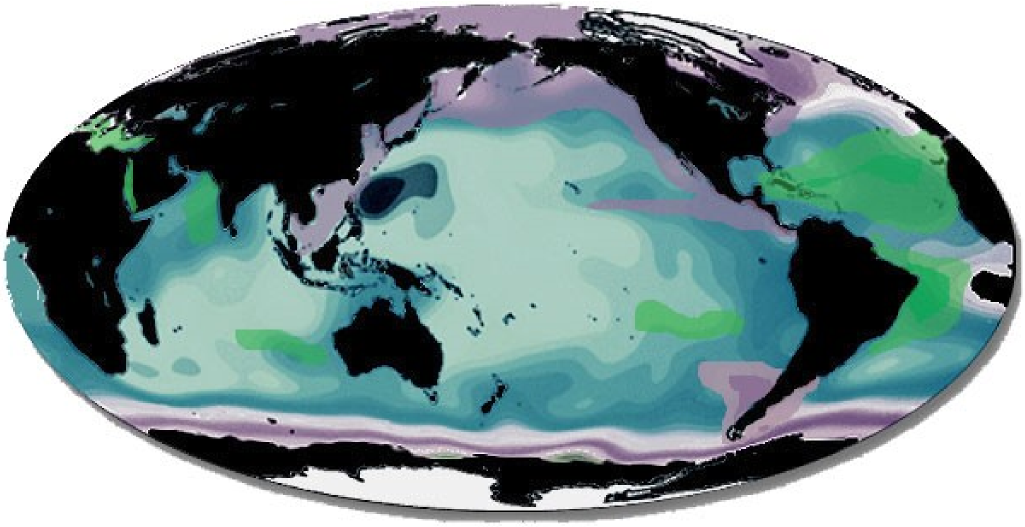

Figure 2.4: Salinity map showing areas of high salinity (3.6%) in green, medium salinity in blue (3.5%), and low salinity (3.4%) in purple (Marine Science 2008)

From Figure 2.4 the average salinity of sea-water around Australian is 35 parts per thousand. Therefore to simulate this environmental condition the water would consists of

3.5% dissolved salts.

The effect of sea-water exposure has been tested by Reis (2009) and is presented in Table 2.1. The test focused on DGEBA based epoxy resin with filler, however, the samples were post-cured at 80oC for 3 hours instead of ambient temperatures. From Table 2.1 the results indicate excellent chemical resistance. The effect of sea-water on the durability of the specimens was slightly greater than distilled water. It was mentioned in Section 2.2.2 that exposure to distilled water is unlikely, therefore, using tap water may present different results.

Table 2.1: Flexural and Compressive Strength of Polymer Concrete after Degradation Cycles (Reis 2009)

Solution Type	**PH**	**Flexural Strength MPa**	**% Strength % Loss**	**Compressive Strength MPa**	**% Strength % Loss**
Reference		24.73	-	51.93	-
Distilled water	5.1	23.36	5.9	51.55	0.7
Soft drink	2.6	24.57	0. 6	38.13	36.2
Sulphuric acid	0.1	22.16	11.6	51.49	0.9
Seawater	8.1	23.03	7.4	49.14	4.7
Lactic acid	1.9	29.66	25.8	40.34	28.7
Citric acid	2.0	22.36	10.5	49.75	4.4
Formic acid	1.9	3.78	84.7	23.07	55.6
Acetic acid	2.5	14.72	68.1	48.22	7.1

Now that environmental conditions have been identified the properties of epoxy resin will be discussed.

2.3 Epoxy Resin

To mitigate the effect of concrete disadvantages Polymer Concrete (PC) is the most conventional method used for concrete crack repair and coating (Lokuge & Aravinthan 2013, Muthukumar & Mohan 2004, Reis 2009). Epoxy is a type of PC as can be seen in Figure 2.5.

The most commercially available and widely used epoxy resin, and the one that will be experimented with, is diglycidyl ether of bisphenol-A (DGEBA) (Chruciel & Leniak 2015).

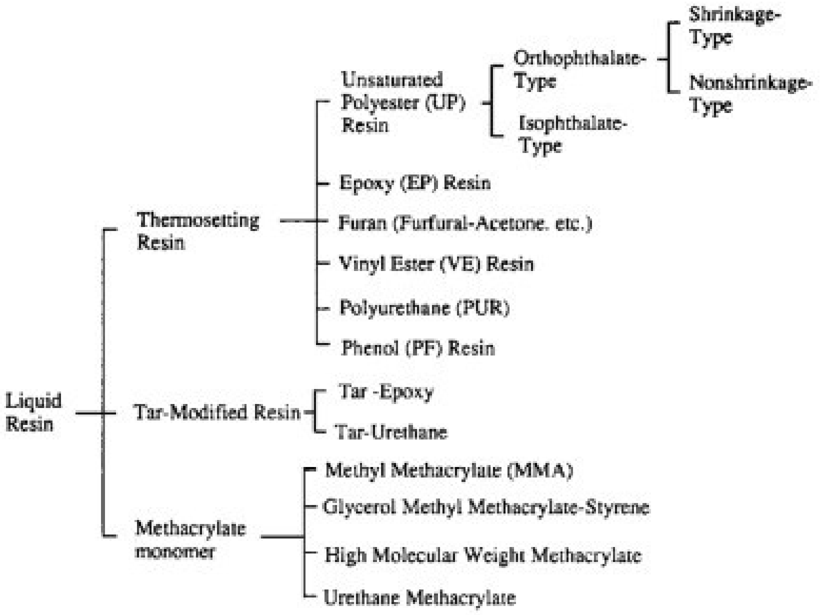

Figure 2.5: Classification of liquid resins for polymer mortar and concrete (Ohama 1997)

The molecular structure of DGEBA is shown in Figure 2.6.

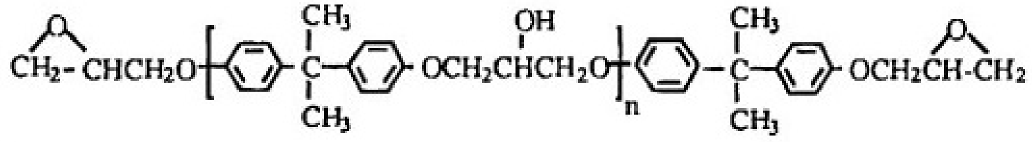

Figure 2.6: The molecular structure of DGEBA (Chruciel & Leniak 2015)

According to Kirlikovali (1981) the average mechanical properties of PC are presented in Table 2.2 below.

Table 2.2: Properties of Concrete-polymer Composites in Comparison with Ordinary Concrete (Kirlikovali 1981)

Property	**PC**	**Concrete**
Compressive strength, psi	20,000	5,000
Tensile strength, psi	1,400	250
Modulus of elasticity, 10^6 psi	5.3	3.6
Shear strength, 10^3 psi	3.7	1.1
Modulus of rupture, $10^3$3 psi	2.2	0.7
Coefficient of expansion, 10^{-6} in./in.-°F	5.3	4
Water permeability, 10^{-4} ft/yr	0	
Water absorption, %w	0.3	5.3
Freeze/thaw resistance		
# of cycles	3,300	590
% weight lost	0	25
Hardness, impact hammer	55	32
Acid resistance		
% weight lost after 3 months of immersion		
5% HCI	0.3	24
15% HCI	3	27
10% H2SO4	1.2	39
Sulphate attack		
% expansion after 2 years of exposure	0.003	0.5
Corrosion by distilled water	None	Severe
Thermal conductivity		
BTU/ft^2-h-°F	1.206	1.332

When comparing the properties of concrete to the polymer concrete, it can be seen why it is commonly used to coat concrete (Lokuge & Aravinthan 2013, Muthukumar & Mohan 2004, Elalaoui, Ghorbel, Mignot & Ouezdou 2012, Reis 2009).

When it comes to the curing time of the epoxy resin, there are inconsistencies within the literature. Lokuge & Aravinthan (2013) claims 80% of 28-day compressive strength is reached after 7 days. Bedi, Chandra & Singh (2013) on the other hand states that PC establishes 70-75% of its compressive strength curing at ambient temperature for 24 hours. At 7 days the gain of compressive strength is claimed to be negligible. For this report the specimens will be cured for 7 days before testing to ensure adequate strength is reached.

According to Chruciel & Leniak (2015) majority of current research about epoxy resin has been focused on improving mechanical properties, thermal stability, flame resistance and raising its glass transition temperature (Tg). The effect of temperature is mentioned next.

2.3.1 Temperature

Temperature has a great influence on the mechanical properties of epoxy resin. The two commonly known effects are within the curing process and after the samples have been cured.

Elalaoui et al. (2012) states that little research has been done into the effect of temperature. Studies have commonly focused on the effect of temperature on an already optimised PC mixture. This project instead focuses on finding the optimised mixture with respect to the effect of temperature.

CURING

Temperature during the curing process can affect the mechanical properties of the final epoxy concrete. It can alter the duration of the curing process, and it is recommended that epoxy is not cured under 10°C due to significant deceleration (Michels et al. 2015).

Epoxy is usually cured at ambient temperatures for economical reason, though within the temperature range 70-100°C the duration of curing is reduced (Michels et al. 2015). Custdio et al. (2011) suggests that curing at ambient temperatures only partially cures samples. This would increase potential for shorter life span, especially at elevated temperature. This theory was only hypothesised at the publication of the literature, therefore, the possible implication it has on the experimentation is ignored. This would be a recommendation for further testing.

CURED SAMPLES

Fully or partly cured epoxy specimens are prone to lose in mechanical properties when subjected to increased temperature(Michels et al. 2015, Elalaoui et al. 2012, Zhou & Lucas 1999). The effect is also known as the glass transition range and is the transition from solid to rubber-like state. The process is a continuous effect over a certain range as can be depicted from Figure 2.7. The glass transition temperature (Tg) is marked out in the figure.

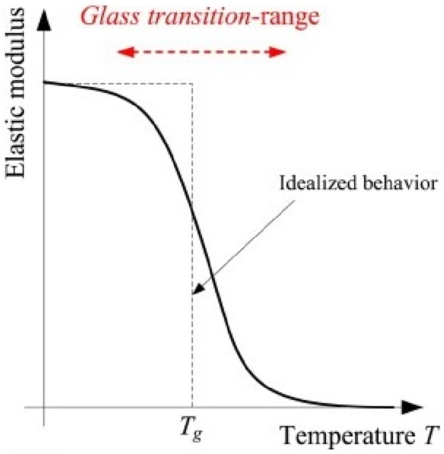

Figure 2.7: Schematic elastic modulus loss of an epoxy resin with increasing temperature (Michels et al. 2015)

The glass transition temperature is not a defined material property, due to it varying with curing and testing parameters (Michels et al. 2015). According to Zhou & Lucas (1999), Tg is also influenced by hygrothermal environments. Due to it not being a defined material property commercial product data often excludes the Tg (Custdio et al. 2011). Therefore it has to be obtained from either from the manufacturer or experimentally. Even when the information is obtained from the manufacturer, it is not stated how the value was obtained.

While it sometimes affected ambient temperature performance, Custdio et al. (2011) also concluded that post-curing treatment to epoxy specimens lead to higher Tg than other temperature treatments. Where post-curing is the process of subjected the specimen to elevated temperature for a set duration, allowing to return to ambient temperatures before being tested.

2.4 Filler

The addition of light-weight particulate fillers in the PC is to minimise the detrimental effect of temperature on the epoxy and make it more economical. Generally the most economical epoxy mixture contains minimal amount of polymer, due to the expense of the polymer (Lokuge & Aravinthan 2013). Fillers are also usually particles of a size less than 80 microns. This can increase strength due to a reduction in void content (Bedi et al. 2013).

In this project the fillers used are fly ash, hollow microspheres and fire retardant. This section focuses on the published works that have focused on the effect filler has had on the thermomechanical properties of epoxy resin.

Jin & Park (2012) conducted a study focused on determining the effect filler material had on the Tg. This is similar to the focus that this report, however, there are some major differences. Firstly the filler material considered was nano-Al2O3 particles and nano-SiC particles. These particle arent conventionally used, so werent considered in this report. Secondly the sample were cured at temperatures above 100°C. This is not practical with respect to the desired structural applications. The results of the study are shown in Figure 2.8.

From these results it can be noted that while the filler didnt significantly change the Tg, it did improve the effect the temperature had on the mechanical properties past the Tg.

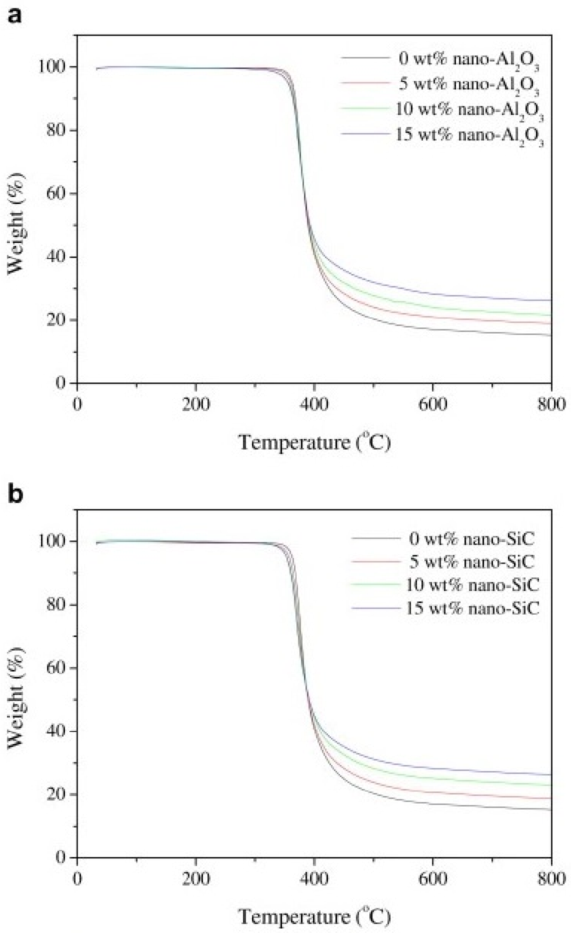

Figure 2.8: The Effect of Temperature on Composites of (a) nano-Al2O3 and (b) nano-SiC (Jin & Park 2012)

2.5 Summary

This chapter analysed literature needed to determine the effect of elevated temperature and simulated environmental conditions on the properties of PFR for structural applications. From the published literature the environmental conditions epoxy is exposed to have been identified. Those environments are air (control), water, salt-water and hygrothermal. It was also determined that epoxy has a high sensitivity to temperature.

From the published work, one major gap in the research was noticed. That was that little research had been done to determine if filler material could improve thermomechanical properties of epoxy resin. For the research that had been perform the specimens had been post-cured. It was mentioned in Chapter 1 that, post-curing was uneconomical and inconvenient for large concrete structures. This lead to the main motivation of this study, to analyse the effect light-weight particulate filler has on the thermomechanical and durability properties of epoxy resin cured at ambient temperatures.

The following chapter focuses on the experimentation and methodologies to be used based on the implication of the research.

Chapter 3

Methodology

3.1 Introduction

This chapter outlines the methodology of the two studies with respect to the findings in Chapter 2, including the project planning.

3.1.1 Study 1 - Temperature Effect

The aim of Study 1 is to meet the first design objective:

Determine the optimal filler content, with respect to elevated temperature, that had no significant reduction in compressive strength of the epoxy resin

To satisfy this aim, Study 1 will test the following epoxy resin to particulate filler ratios; 100:0, 80:20, 60:40 and 40:60. The samples will also be tested at the following temperatures; Room temperature (23°C), 40°C, 60°C, 80°C and 100°C. The optimal filler content will have no significant reduction on the compressive strength with the increase of the temperature, when compared to initial results at room temperature.

The properties investigated in Study 1 are physical observations and compressive strength.

3.1.2 Study 2 - Environmental Exposure

The aim of Study 2 is to meet the second design objective:

Evaluate the six month durability of the optimal mix design with respect to simulated environmental conditions.

To satisfy this aim, Study 2 will expose the samples to air, water and saltwater environments for six months, approximately 168 days, and a hygrothermal environment for one month. The samples will be tested under compression at predetermined intervals.

The properties investigated in Study 2 are physical observation, compressive strength, microscopic observation, weight/absorption, and dimension/shrinkage/swelling.

3.2 Project Planning

3.2.1 Timeline

Table 3.1 shows the timeline of various phases of the report. The dates shown are for 2015 and planned dates werent strictly adhered to.

Table 3.1: Timeline

Task	**Planned Date Range**	**Actual Date Range**
Literature Review	March - July	March -
Methodology	Jan - Feb	Jan - Feb
Experiment Preparation	Jan - Feb	Jan - Feb
Study 1 Experiment	Jan	Jan
Study 2 Experiment	Feb - July	Feb - July
Analysis and Interpretation	March - Aug	April - August
Discussion and Recommendations	May - July	June - August
Conclude the Results	Aug - Sep	October

The main causes for the timeline not being adhered to were personal health, work and study commitments.

Another timeline that has been provided is for the preparation and compression testing for Study 1 and 2. This is located in Table 3.2. To maintain accuracy if a testing date was missed, testing should be performed at the next applicable date. The new date would be noted and graphical results would reflect the actual date of testing.

Table 3.2: Testing Timeline

Task	**Dates**
Preparation of Sample - Study 1	8-9 January
Study 1 - Compression Testing	16 January
Preparation of Samples Study 2	3-6 February
Study 2 - Initial Compression Testing	10 February
Study 2 - Hygrothermal 1 Day Testing	11 February
Study 2 - Hygrothermal 3 Day Testing	13 February
Study 2 - 7 Day Compression Testing	17 February
Study 2 - 28 Day Compression Testing	10 March
Study 2 56 Day Compression Testing	7 April
Study 2 112 Day Compression Testing	2 June
Study 2 168 Day Compression Testing	28 July

3.2.2 Resource Requirements

The resources required for the Study 1 and 2 are available at University of Southern Queensland (USQ) and the Centre of Excellence in Engineered Fibre Composite (CEEFC). Relevant staff and the supervisor were consulted about access to the materials and test equipment for use under relevant supervision. Sample preparations were performed at P11, while access to the machines used for testing were available at P9. Access to the optical microscope used for microscopic observation was available in Z block.

3.2.3 Safety

Safety aspects of the project were required for the physical preparation and testing of the samples. Personal protective equipment (PPE) and assistance was provided by trained CEEFC staff. The risk assessment is provided in Appendix B Risk Assessment. The following PPE were used at various stages:

- Steel Capped Boots (Personally Owned)
- Disposable Coveralls
- Gloves
- Safety Glasses
- Comfort Mask

3.3 Preparing Samples

The PFR consists of the five following materials; epoxy resin type DGEBA (Part A), amine based curing agent (Part B), Hollow Microsphere (HM), Fire Retardant Filler (FRF) and fly ash. A mixing ratio of 100g Part to 32g Part B is required for the resin mix to be reactive. The mixing formulation for the Light-Weight Particulate Filler used in this study was established by CarbonLoc and due to commercial confidentiality, could not be included in this report. The materials are shown in Figure 3.1 and 3.2 below.

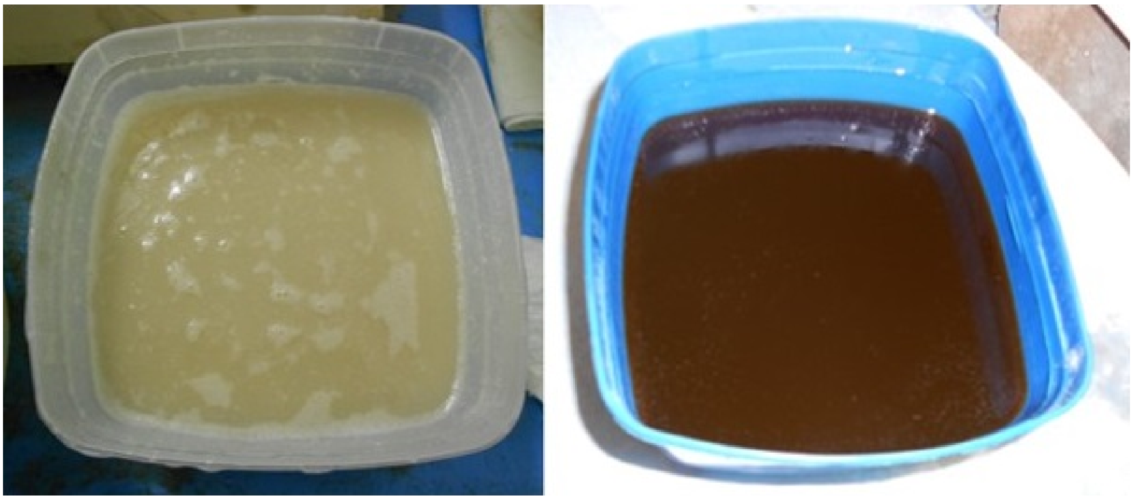

Figure 3.1: DGEBA Resin and Amine Based Curing Agent Respectively

Figure 3.2: Hollow Microsphere, Fire Retardant and Fly Ash Respectively

One inch nominal PVC pipe was cut for use as the sample moulds, due to ease of supply and procurement for multiple samples. The PVC pipe met the required 25mm by 25mm compression testing standard as provided by ASTM International C579 which was reapproved in 2012. The PVC pipe was sealed at one end with duct tape and sticky tape as it ensured easy removal for demoulding.

Using the volume of the individual moulds, the equivalent weight of each material was calculated for each PFR mix. The mix proportions for individual samples are provided in Table 3.3. In the preparations for each study the mix proportions were multiplied for samples required with additional 5%.

Table 3.3: Mix Proportions of PFR per Sample

Resin : Filler (by volume)			**Mix 1** 100:0	**Mix 2** 80:20	**Mix 3** 60:40	**Mix 4** 40:60
Resin	Part A	grams	17.57	14.05	10.54	7.03
	Part B	grams	5.62	4.5	3.37	2.25
Filler (HM + FRF + Fly Ash)		grams	0	8.38	16.75	25.13

The sample size for each tested scenario was three, any additional samples were tested at initial conditions. Table 3.4 presents the samples required for Study 1. The resin to filler ratio is tested at four increments of 20% and the temperature is tested at four increments of 20°C and at room temperature.

Table 3.4: Samples Required for Study 1

Resin/Filler Ratio		**100:0**	**80:20**	**60:40**	**40:60**
Temperature (°C)	23	3	3	3	3
	40	3	3	3	3
	60	3	3	3	3
	80	3	3	3	3
	100	3	3	3	3
Sub Total		15	15	15	15

Table 3.5 presents the samples required for Study 2. The samples were exposed to one of four different environments; the control air, sea water, water and hygrothermal. Due to time constraints, the duration of Study 2 was limited to approximately 6 months. To ensure availability for testing, the testing weekday stayed the same, resulting in the duration being expressed in days. Therefore the arbitrary month duration was converted to a four week month.

The hygrothermal environment wasnt tested for the same durations as the other three environments. In Chapter 2 hygrothermal was defined as the combined effects of temperature and moisture. Unlike the other three environments which provide continuous exposure, hygrothermal environments typically dont provided extended continuous exposure, for example humid climates. Due to the inconsistency in exposure it was necessary to test samples after 1 and 3 days and testing cessed after 28 days.

Table 3.5: Samples Required for Study 2

Environment		**Air**	**Sea Water**	**Water**	**Hygrothermal**
Time in Environment	Initial Strength	3	-	-	-
	1 Day	-	-	-	3
	3 Days	-	-	-	3
	7 Days	3	3	3	3
	28 Days	3	3	3	3
	56 Days	3	3	3	-
	112 Days	3	3	3	-
	168 Days	3	3	3	-
Sub Total		18	15	15	12

This resulted in a total of 60 samples for Study 1 and 60 for Study 2.

For the preparation of the samples, full PPE was required to be worn and preparation took place in one of laboratory in P11 at University of Southern Queensland. The epoxy resin materials and the light weight particulate filler materials were mixed separately first. This ensured consistent mixing and allowed the epoxy resin to become completely mixed and reactive before filler was added.

Using a digital scale and a disposable cup, Part A was measured first into a 2L container as can be seen in Figure 3.3. To ensure that material wasnt lost in the changing of containers, the digital scales were tare weighed and Part B was carefully measured into the container. This is illustrated in Figure 3.4.

Figure 3.3: Measuring Part A into 2L Container

Figure 3.4: Measuring Part B into the Container Containing the Pre-Measured Part A

Once both materials had been measured, they were mixed together using a spoon until the resin had a uniform consistency. Figure 3.5 illustrates the materials being mixed together.

Figure 3.5: Epoxy Resin Materials Being Mixed Together

The measuring of the light weight particulate filler materials was the same as the epoxy resin materials, although in an arbitrary order of measuring. Once the three materials had been measured they were mixed until uniform consistency, as can be seen in Figure 3.6.

Figure 3.6: Light Weight Particulate Filler Materials Being Mixed Together

Due to the high viscosity of the epoxy resin mixture, it was more practical to add the filler mixture to the epoxy resin mixture. The filler was gradually folded into the resin to reduce the amount of filler that became airborne in the mixing process. This process is illustrated in Figure 3.7. Once all the filler had been folded in, the PFR mixture was mixed until a uniform consistency was reached.

Figure 3.7: Combining Light Weight Filler and Epoxy Resin

The mixture was then poured into the previously prepared moulds and compacted to minimise voids. To ensure that the samples were not mixed up the moulds were labelled before pouring. The samples were then allowed to cure at ambient temperatures, as illustrated in Figure 3.8.

Figure 3.8: Curing of Samples for Study 1

Between preparations of different epoxy resin to filler ratios, all containers and utensils were cleaned using paper towels and methylated spirits. This ensured that containers were sterile before use and avoided excess material in mixture.

Due to the rapid curing of the epoxy resin, samples were able to be demoulded 24 hours after initial curing. To ensure the samples had reached there optimal strength before testing, the sample were cured for 7 days at ambient temperatures. Demoulding the samples first entitled removing the duct tape sealing one end of the PVC Pipe. Using a vice and a hacksaw the PVC was cut lengthwise to allow the samples to pop out of the mould. Care was taken to ensure the hacksaw minimally cut into the sample. If the sample was still difficult to remove a second cut was place on the opposite side to the original cut. Once the samples were removed, sandpaper was used to flatten the top and bottom of the samples. This ensured that in the compression test, the force would be evenly distributed.

For Study 1 once the samples were 7 day cured, the following properties were investigated; physical observation and compressive strength. The method used to investigate these properties is mentioned in Section 3.4. In Study 2 only initial testing occurred once the samples were 7 day cured. Initial properties investigated included the physical observation, compressive strength and microscopic observation of the initial strength samples in Table 3.5 and the weight and dimensions of all the samples. Using a permanent marker all samples were labelled by environment they would be exposed to a date tested. After the weight and dimensions were measured the additional samples were exposed to their respective environment.

There was a total of four simulated environments for Study 2 including air, water, sea water and hygrothermal. Air was the control environment and entitled placing the samples aside in a well ventilated area. Using tap water, a glass container with a lid was filled for the water environment. The samples were left in the water with the lid was necessary to prevent evaporation of the water. To ensure the water didnt become stagnant the water was changed on either the next testing date or one month. The sea water was the same as the water environment, however, to match Australian costal climates sea salt was used to mix the water to 3.5% salinity. Figure 3.9 shows the samples in the air, water and sea water environments.

Figure 3.9: Samples in Air (Left), Water (Top Right) and Sea Water Environment (Bottom Right)

To simulate a hygrothermal environment the samples were placed in a metal pot with a lid filled with tap water. The lid was necessary not only to prevent evaporation of the water but to insulate the heat in the pot. The pot was placed on a temperature controlled electric stove with the thermometer sensor submerged in the water as can be seen in Figure 3.10. The temperature gauge was set to approximately 60°C as can be seen in Figure 3.11. Unlike the water and sea water environment, the hygrothermal water was not replaced during the month. This was to keep the samples at a constant temperature.

Figure 3.10: Samples in the Hygrothermal Environment

Figure 3.11: Temperature Gauge for Electric Stove

To maintain a relatively consistent exposure the samples were keep in their simulated environment except for changing of the water for the water and sea water environment and testing. The methods used for the property investigation is mentioned in Section 3.4.

3.4 Properties Investigated

Between the two studies a total of five different properties were investigated. They are physical observation, compressive strength, microscopic observation, weight/absorption, and dimension/shrinkage/swelling. This sections explains the method these properties were investigated.

3.4.1 Physical Observation

Physical observations included observations during and after testing and comparison between the compressive failures of the samples. For the comparison between compressive failures the samples were placed together and a photo was taken. Physical observations were investigated for Study 1 and 2.

3.4.2 Compressive Strength

Study 1 and 2 used a 100kN capacity testing machine, located in P9 at USQ, for the compression testing. The results of the compression testing included the peak stress, peak strain and stress vs strain curve. From the stress vs strain curve the modulus of elasticity of the sample could be calculated. Figure 3.12 illustrates one of the samples being tested at room temperature.

Figure 3.12: Epoxy Resin to Filler Ratio 40/60 Being Tested at Room Temperature

For Study 1 two additional pieces of equipment were used to precondition the samples and maintain the temperature whilst testing, these were an oven and environmental chamber respectively. The oven was used to precondition the samples by heating them up to the temperature they were being tested at. Samples were tested in order of increasing temperature and were kept in the oven for approximately half an hour to ensure they were heated through. When the samples were being tested they were removed and taken to the environmental chamber. The next lot of samples were then placed in the oven and the temperature increased.

The environmental chamber was used to encase the compression testing machine for temperature control during the testing. It was set to the temperature being tested and to ensure temperature control the door remained shut when not loading samples. At room temperature the door was left open as temperature control was not required. In Figure 3.12 it illustrates testing at room temperature with the door open. Due to height constraints the bottom plate of the machine had to be raised. Figure 3.13 illustrates the whole environmental chamber around the compression testing machine.

Figure 3.13: Compression Testing Machine with Environmental Chamber Around it

3.4.3 Microscopic Observation

Microscopic observations were performed on post compression tested samples in Z Block at USQ with an optical microscope. The observations were taken of the flat surface of the samples at the strongest magnification of the available optical microscope. This magnification was 50 times and pictures were taken of the magnification for comparison. Comparing the photos of the microscopic observations would determine if extended exposure to the environments had an effect on the epoxy resin matrix.

3.4.4 Weight

The weight of the samples were measured for Study 2 using an analytical balance accurate to 0.0001g. Weighing the samples determined if absorption or degradation occurred in the samples, by recording increases and decreases in weight respectively. To increase the accuracy of the testing excess water was wiped from the surface of the samples with a clean cloth. The samples were then weighed immediately after, to eliminate further drying of the sample.

All the samples were weighed initially before environmental exposure and on the day of the compression testing. They were also weighed on the preceding compression testing day of their own. If the sample was not being tested that day, it was replaced in its environment.

3.4.5 Dimension

The height of the samples were measured for Study 2 using a Digital Calliper. Measuring the dimensions of the sample it can determine if shrinkage or swelling occurred in the samples. To increase the accuracy of the measuring, using the cut mark left in the samples upon demoulding as a guide, the samples were measured in the same place. Dimension was measured at the same time as weight.

3.5 Summary

This chapter presents the experimental program for the two studies conducted in this thesis. The details of the specimens, how they are prepared and the test procedures were presented.

The specimens consisted of epoxy resin type DGEBA (Part A), amine based curing agent (Part B), Hollow Microsphere (HM), Fire Retardant Filler (FRF) and fly ash. Using P11 at University of Southern Queensland, the materials were mixed together and placed in PVC moulds. They were then cured for 7 days at ambient temperature. Upon removing from the moulds, the specimens were able to be tested.

Study 1 consists of specimens varying from 100:0 to 40:60 epoxy resin to light weight filler. The samples were then be exposed to increasing temperature, from room temperature to 100°C, during the compression testing. From the properties investigated, physical observations and compression testing, the optimal mix design will be determined.

Study 2 consists of the optimal mix design being exposed to simulated environments for a duration of 6 months. The environments included air, water, salt-water and hygrothermal. For Study 2, five properties were investigated; physical observation, compressive strength, microscopic observation, weight and dimension. From the results the durability of the optimal mix design will be determined.

The results, observations and analysis from Studies 1 and 2 are presented in Chapter 4 and 5, respectively.

Chapter 4

Effects of Elevated Temperature on Compressive Behaviour of Epoxy-Based Polymer Resin

4.1 Introduction

This section presents the results and observations as well as the discussion on the compressive behaviour of epoxy-based polymer matrix at elevated temperature. In Chapter 3, the preparation of the epoxy based polymer matrix with different percentages of epoxy resin and light-weight particulate filler was presented. These specimens were then tested under compression in an environmental chamber at room temperature, 30, 40, 60 and 80°C. The physical observations, load-deformation relationship and failure mechanisms of the polymer matrices with different percentages of fillers are presented. Analysis and discussion on the effects of elevated temperature on the parameters considered in this study are also presented.

4.2 Results and Observations

The collected data from the testing is provided in Appendix C. The results and observations are presented based on epoxy to filler ratio. Analysis and discussion with respect to the effect of temperature is presented in Section 4.4.

4.2.1 Specimen 100:0

Physical Results

Physical observations were made to all specimens. The observations noticed for epoxy to filler ratio 100:0 are illustrated in this section and are separated into observations during and post compression testing.

DURING TESTING

The first observation was the quality of the polymer concrete samples. The 100% epoxy samples were translucent and white lumps were observed at the bottom of the sample. After the compression test sticky residue had seeped out of the end where the white lumps were present as can be seen in Figure 4.1. Upon compression, the bottom end widened significantly more than the top end. For samples tested in inverted position, the end with the white lumps would still widen further. This was evident across the temperature range. The failure in the 100% epoxy resin samples resulted in the wider end fractured outwards in a circular manner (Figure 4.3).

Figure 4.1: 100% Epoxy Sample After Compression Testing

The second observation was in relation to the compressive properties of the epoxy resin at higher temperature. Compared to Portland cement, the epoxy resin has higher Youngs modulus values (Kirlikovali 1981), however, the polymer matrix is susceptible to temperatures within the glass range (Michels et al. 2015, Elalaoui et al. 2012, Zhou & Lucas 1999). Glass transition range as previously mentioned is when the polymer state transitions between a solid and rubber-like state with the effects of temperature. In the rubber-like state the polymer matrix is weakened and the elasticity is increased. The higher elasticity would allow the sample to be deformed under the load and return to its original shape upon unloading.

The second observation illustrated this property in the 100% epoxy resin samples. In Figure 4.2 the 100% epoxy resin sample was loaded and then unloaded at 60°C. With the increase in temperature the sample was easily deformed under the load and returned to its original shape upon unloading. From this observation it can be confirmed that the epoxy reacted as expected and the 100% epoxy resin was a suitable control sample.

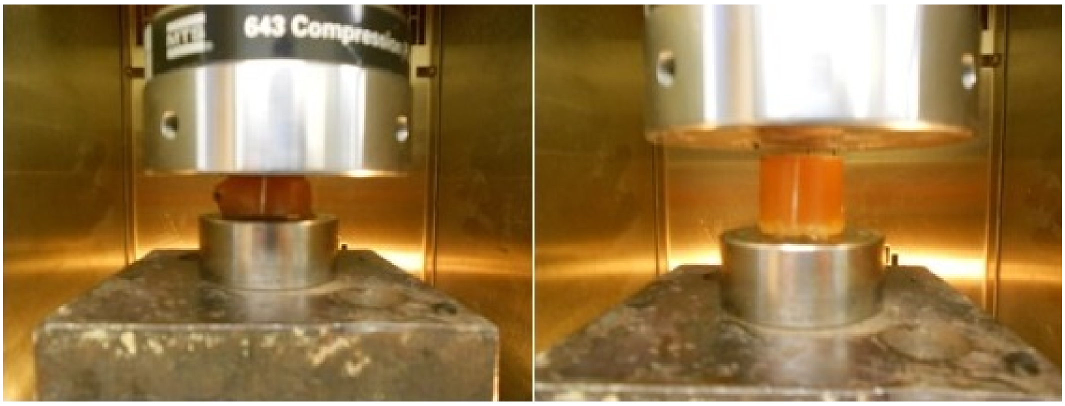

Figure 4.2: Compression Test of 100% Epoxy Resin at 60°C

The final observation was related to the compression testing temperature range. In Chapter 3 it was mentioned that originally the experiment was planned to test temperatures up to 100°C. Due to the compressive failure of the 100% epoxy resin samples at 80°C it was decided that in would be more beneficial to test the samples 30°C instead of the originally planned 100°C. This resulted in two irregularities in the following figures.

Firstly the samples tested at 30°C had 100 written on top instead of the temperature they were tested at. Secondly there was excess heat present in the compression testing machine, after testing the 80°C samples, when initial testing of the 30°C samples started. This resulted in the first two tested samples of 100% epoxy resin heating up and compressive failure reflected samples at 80°C. The failure was completely different to the samples tested at room temperature and 40°C.

Consequently that compression testing data for those two tests were excluded from trend analysis and the rest of the samples were tested the next working day to ensure that the compression testing machine had dropped below the required temperature. It also explains why in Figure 4.3 the side view of the 100% epoxy resin at 30°C sample has a similar compression failure as the 100% epoxy resin samples at 80°C.

POST TESTING

During the compression testing it was observed that the epoxy resin samples reacted as expected to the increasing temperature. At 80°C, however, it appeared that the elastic deformation limit was reached. Unlike the samples at the lower temperatures which were minimally deformed after unloading, at 80°C the samples fractured. This can be seen in Figure 4.3 as the sample to the far right show signs of splitting down the centre and the weaker end, as mentioned in observation one, crumbled.

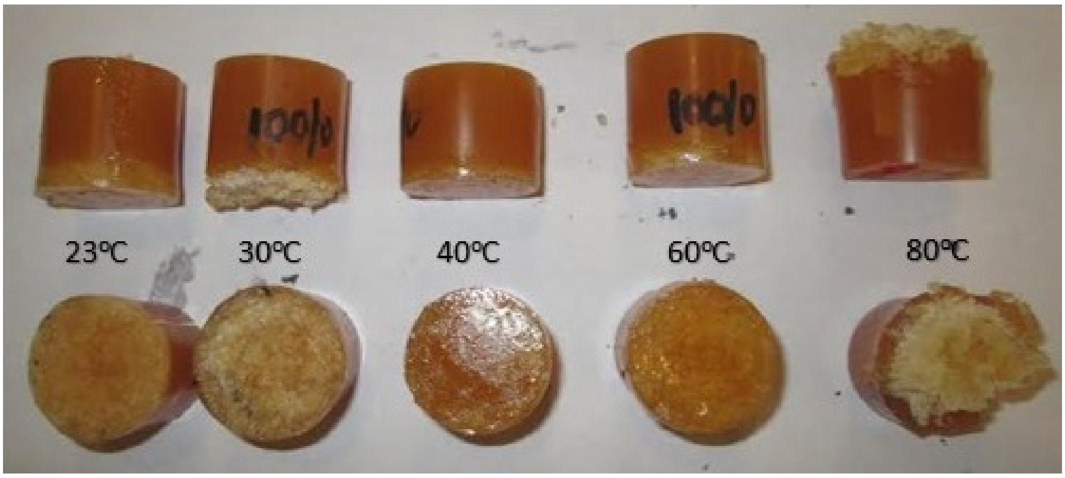

Figure 4.3: 100:0 Epoxy to Filler Ratio Compression Samples at Increasing Temperatures

Figure 4.3 shows the compression tested sample of the 100% epoxy over increasing temperature. This illustrates how the compressive failure progressed with temperature. With the increase in temperature the modulus of elasticity appears to have decreased as the samples sustained greater deformation before compressive failure.

Stress-Strain Behaviour

Figure 4.4 shows the stress-strain behaviour under the compression testing on the 100

The stress-strain relationship curve shows that the behaviour is almost linear for the sample tested at room temperature up to 44 MPa stress and then became non-linear

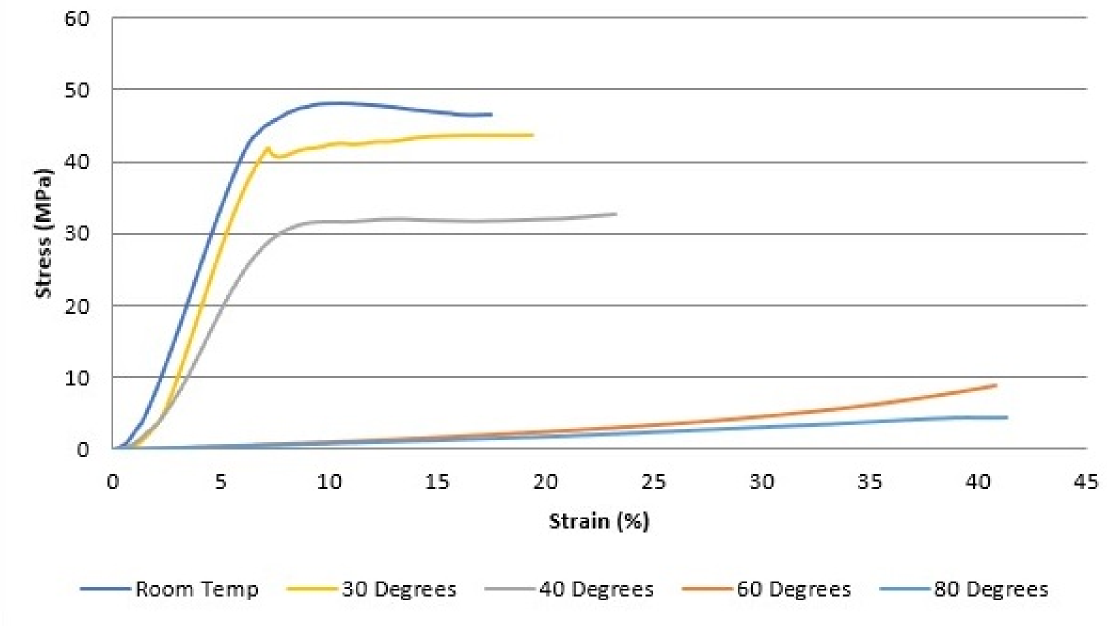

Figure 4.4: Stress vs Strain Graph for 100:0 Epoxy Resin to Filler Ratio

after that. The sample tested at 40°C shows the same trend, however, it is at 25 MPa that the curve becomes non-linear. Samples tested at 60°C and above do not show this trend as the stress strain relationship curve remains relatively linear for the whole test. This resulted in there not being a clear peak stress for the samples tested at 60°C and 80°C.

Table 4.1 contains the average peak stress and Youngs Modulus. Youngs Modulus was determined by plotting only the slope of the stress vs strain graph in excel. A linear trend line was then applied and from the equation of the line the slope was converted to MPa. From the data a decreasing trend was observed with the increase of temperature for both parameters.

Table 4.1: Modulus of Elasticity for 100:0 Epoxy to Filler Ratio

Temperature °C	**Peak Stress MPa**	**Youngs Modulus MPa**
23	48.0	852
30	43.8	867
40	10.67	544
60	5.3	20
80	4.1	11

4.2.2 Specimen 80:20

Physical Results

DURING TESTING

With the addition of the light weight filler the samples were opaque, meaning that white lumps were not observed in the samples. Despite not seeing the white lumps, however, the 80:20 samples failed in a similar way to the 100% epoxy samples. After the compression test sticky residue had seeped out, especially towards the end that failed. The end that failed, like the end that failed in the 100% epoxy samples, had widened significantly more than the top end. The end would fracture outwards in a circular manner. These physical observation can be seen in Figure 4.5. This similarity in compressive failure suggests that the white lumps were also present in the 80:20 epoxy samples.

It was mentioned in Section 4.2.1 that the samples were tested at 30°C instead of the planned 100°C, therefore in Figure 4.5 the samples tested at 30°C had 100 written on top instead of the temperature they were tested at.

POST TESTING

Figure 4.5 shows the compression tested sample of the 80:20 epoxy over increasing temperature. This illustrates how the compressive failure progressed with temperature. With the increase in temperature the modulus of elasticity appears to have decreased as the samples sustained greater deformation before compressive failure. This can be seen as the samples at 40°C and below sustained a permanent crushing failure mechanism. The samples tested above 40°C, on the other hand, experienced a bulging failure mechanism. At the point of failure the bulging resulted in fracturing along the samples, and upon unloading the bulging receded.

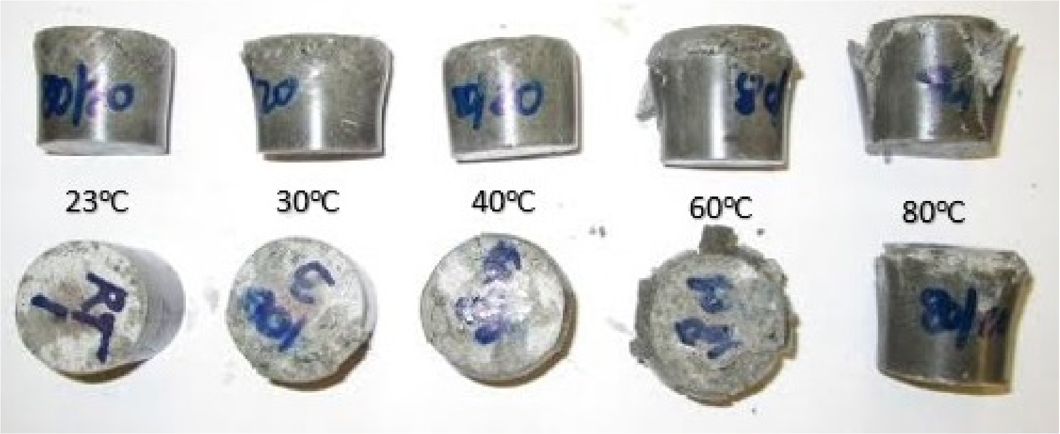

Figure 4.5: 80:20 Epoxy to Filler Ratio Compression Samples at Increasing Temperatures

Stress-Strain Behaviour

Figure 4.6 shows the stress-strain behaviour under the compression testing on the 80:20 epoxy specimens.

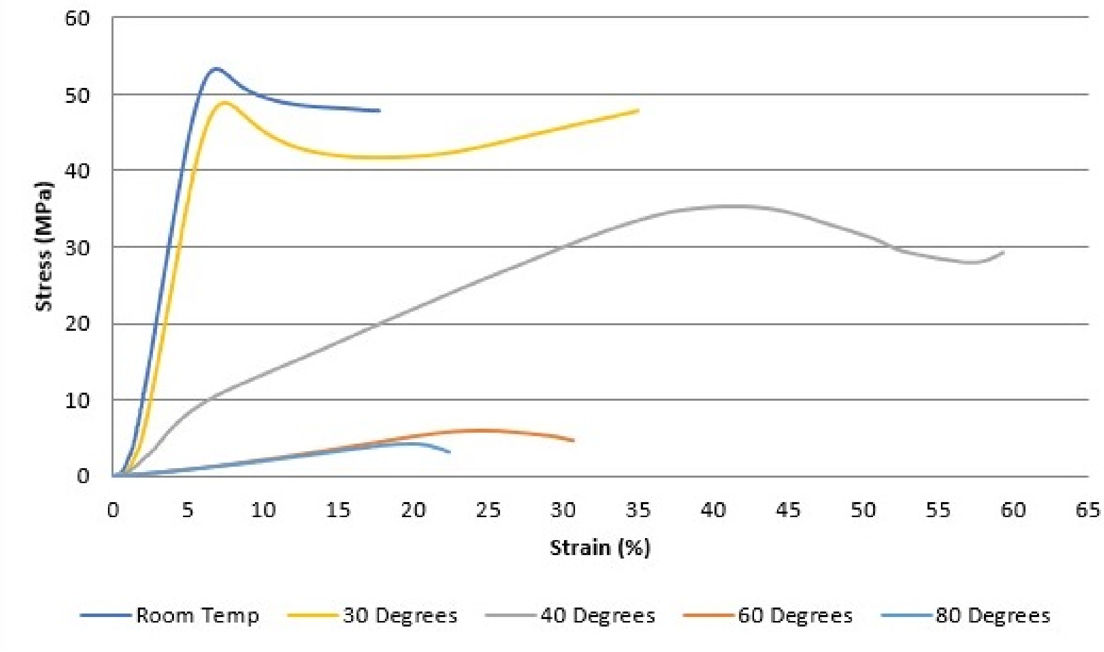

Figure 4.6: Stress vs Strain Graph for 80:20 Epoxy Resin to Filler Ratio

The stress-strain relationship curves in Figure 4.6 are similar to the relationship curves in Figure 4.4. This similarity was especially noticed for the specimens tested at room temperature and 30°C. Unlike the 100% epoxy specimens, for the specimens tested at 60°C and 80°C the compression testing resulted in less deformation before compressive failure. The yield point was also clearly present when compared to Figure 4.4. The specimens tested at 40°C, however, illustrated a completely different trend. It was a combination between the lower and higher temperature trends, with high deformation under high stress.

Table 4.2 contains the average peak stress and Youngs Modulus. From the data a decreasing trend was observed with the increase of temperature for both parameters.

Table 4.2: Modulus of Elasticity for 80:20 Epoxy to Filler Ratio

Temperature °C	**Peak Stress MPa**	**Youngs Modulus MPa**
23	55.4	1124
30	44.1	1002
40	34.0	85
60	6.4	27
80	4.0	23

4.2.3 Specimen 60:40

Physical Results

DURING TESTING

The first observation was in relation to the quality of the polymer concrete samples. The observation was the same noticed in the 80:20 epoxy sample, suggesting that white lumps were also present in the samples. The only difference in the observation was that while the sticky residue was still present, there was no clear weaker end. This suggests that while the white lumps may have been present, due to the higher density of the epoxy mixture they didnt settle to the bottom.

It was mentioned in Section 4.2.1 that the samples were tested at 30°C instead of the planned 100°C, therefore in Figure reffig:4.7 the samples tested at 30°C had 100 written on top instead of the temperature they were tested at.

POST TESTING

During the compression testing it was observed that the epoxy resin samples didnt react like the 100% and 80:20 epoxy samples did with increasing temperature. There wasnt a clear increase in deformation with temperature. Instead the samples had shear cracks along the outer surface, with at 80°C only one massive one was present.

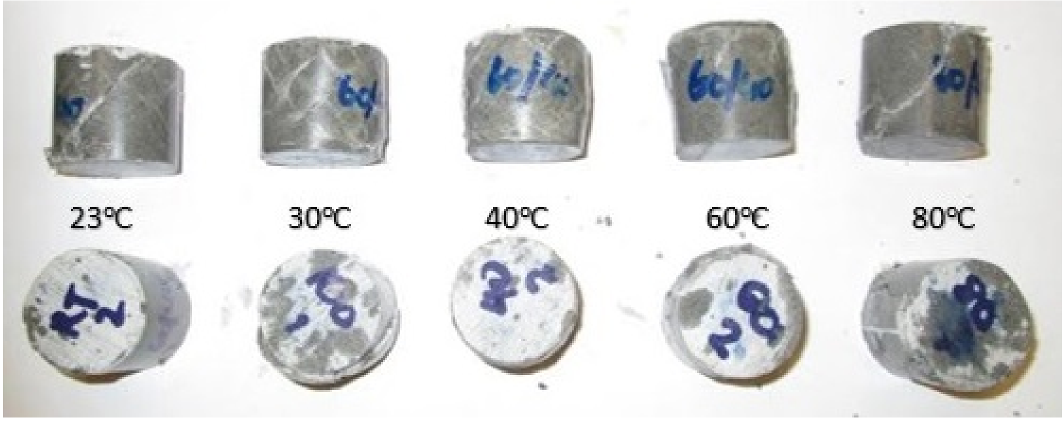

Figure 4.7: 60:40 Epoxy to Filler Ratio Compression Samples at Increasing Temperatures

Figure 4.7 shows the compression tested sample of the 60:40 epoxy over increasing temperature. This illustrates how the compressive failure progressed with temperature. With the increase in temperature the modulus of elasticity appears to have decreased as the samples sustained greater deformation before compressive failure.

Stress-Strain Behaviour

Figure 4.8 shows the stress-strain behaviour under the compression testing on the 60:40 epoxy specimens.

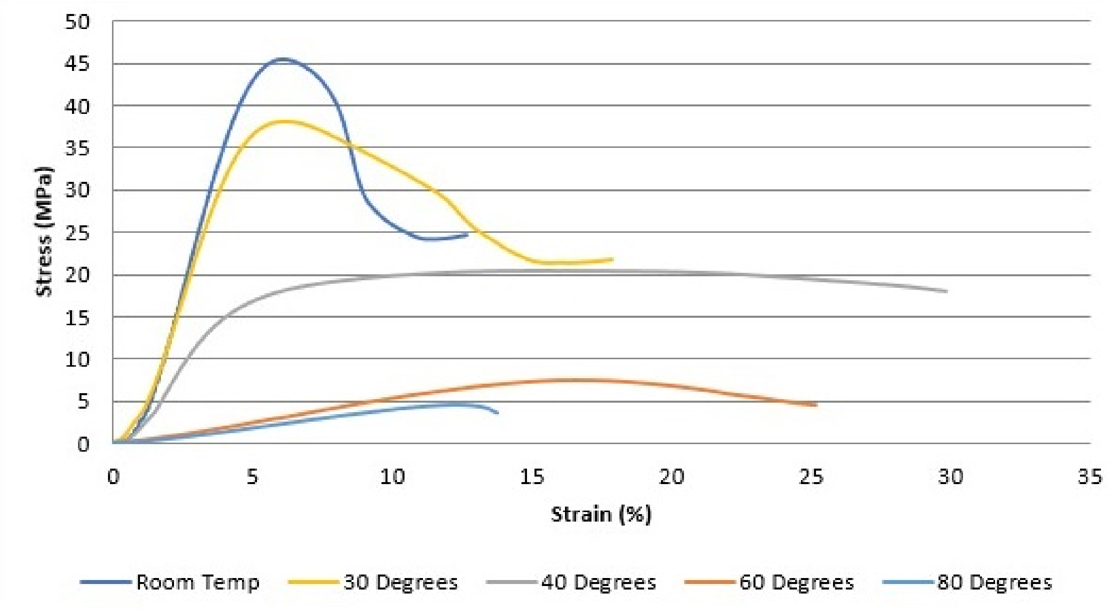

Figure 4.8: Stress vs Strain Graph for 60:40 Epoxy Resin to Filler Ratio

The stress-strain relationship curves in Figure 4.8 are similar to the relationship curves in Figure 4.6. There is a clear difference between the room temperature and 30°C curve, as the later had a lower peak stress. For the specimens tested at 60°C and 80°C the stress-strain curve showed the same trend as in Figure 4.6, however, there was greater deformation before compressive failure. The specimens tested at 40°C, however, illustrated a trend similar to the one seen in Figure 4.4. The peak stress was not large, although, it extended across large deformation.

Table 4.3 contains the average peak stress and Youngs Modulus. From the data a decreasing trend was observed with the increase of temperature for both parameters.

Table 4.3: Modulus of Elasticity for 60:40 Epoxy to Filler Ratio

Temperature °C	**Peak Stress MPa**	**Youngs Modulus MPa**
23	45.1	1154
30	37.8	930
40	21.2	441
60	7.8	56
80	4.4	41

4.2.4 Specimen 40:60

Physical Results

During the preparation of the 40:60 epoxy samples the workability of the samples was almost unworkable. This resulted in difficulty in sample preparation and voids were observed in the samples upon de-moulding.

DURING TESTING

The first observation was in relation to the quality of the polymer concrete samples. The observation was the same noticed in the 60:40 epoxy sample, suggesting that white lumps were also present in the samples though evenly distributed.

It was mentioned in Section 4.2.1 that the samples were tested at 30°C instead of the planned 100°C, therefore in Figure 4.9 the samples tested at 30°C had 100 written on top instead of the temperature they were tested at.

POST TESTING

During the compression testing it was observed that the epoxy resin samples reacted like the 60:40 epoxy samples did with increasing temperature. There wasnt a clear increase in deformation with temperature. Instead the samples had shear cracks along the outer surface for all temperatures tested.

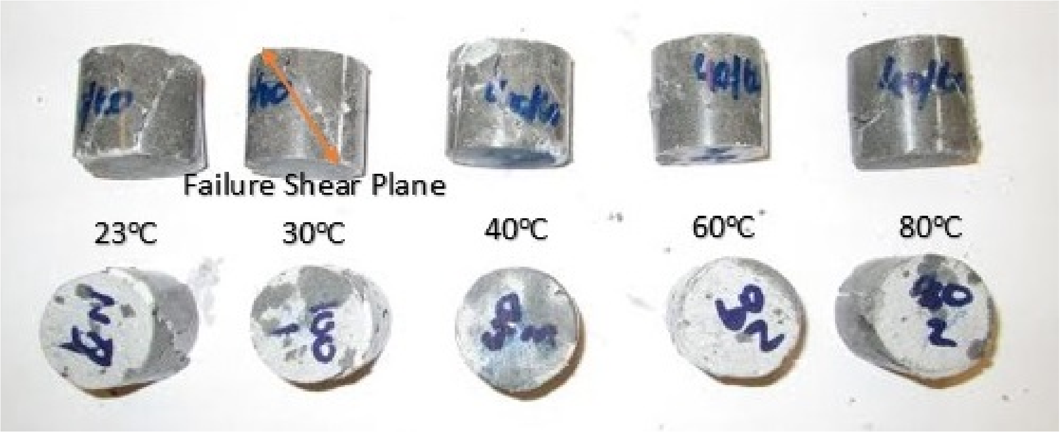

Figure 4.9: 40:60 Epoxy to Filler Ratio Compression Samples at Increasing Temperatures

Figure 4.9 shows the compression tested sample of the 40:60 epoxy over increasing temperature. This illustrates how the compressive failure progressed with temperature. With the increase in temperature the modulus of elasticity appears to have decreased as the samples sustained greater deformation before compressive failure.

Stress-Strain Behaviour

Figure 4.10 shows the stress-strain behaviour under the compression testing on the 40:60 epoxy specimens.

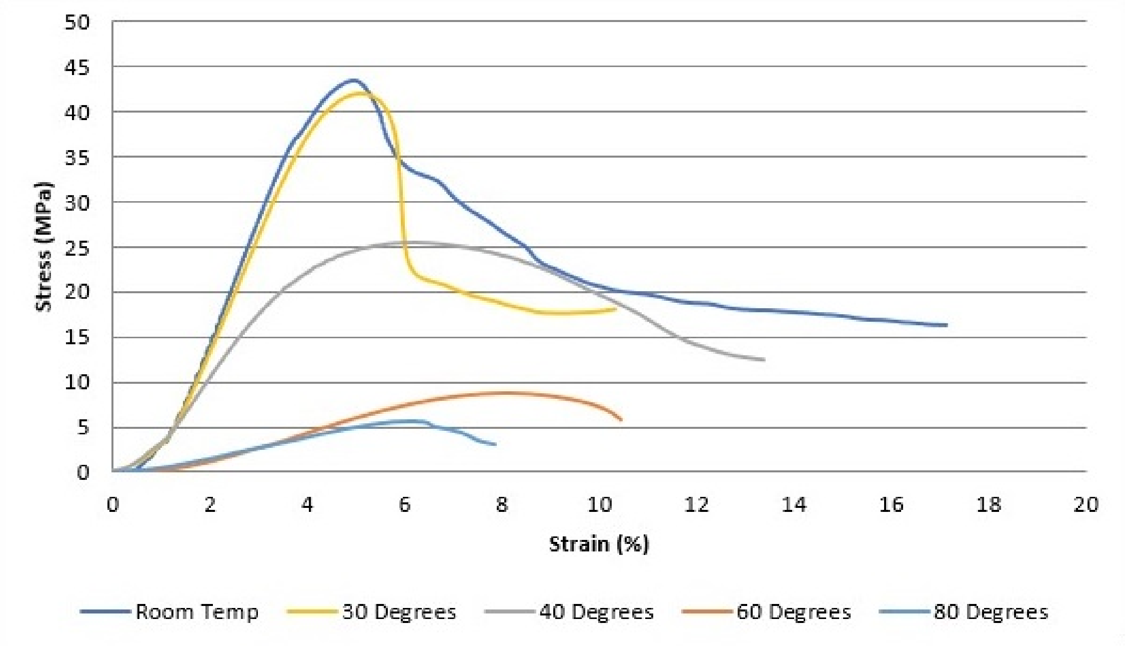

Figure 4.10: Stress vs Strain Graph for 40:60 Epoxy Resin to Filler Ratio

The stress-strain relationship curves in Figure 4.10 are similar to the relationship curves in Figure 4.8. The major difference is that the curves have less of a smooth trend. The room temperature and 30°C curves also have a similar trend and peak stress.

Table 4.4 contains the average peak stress and Youngs Modulus. From the data a decreasing trend was observed with the increase of temperature for both parameters.

Table 4.4: Modulus of Elasticity for 40:60 Epoxy to Filler Ratio

Temperature °C	**Peak Stress MPa**	**Youngs Modulus MPa**
23	36.9	1371
30	38.7	1249
40	25.0	707
60	8.9	140
80	5.5	108

4.3 Discussion

4.3.1 Effects of Filler

This section analyses the effects of temperature for each filler percentage.

Specimen 100:0

With the increase in temperature a couple of trends were noticed for the 100:0 specimens. Firstly it was the behaviour of the stress-strain relationship curve with increasing temperature. Figure 4.11 below illustrates the stress-strain behaviour of different polymers. When compared to Figure 4.4, the samples tested between room temperature and 40°C followed the red curve. The samples tested at 60°C and above, however, followed the green curve. According to Callister & Rethwisch (2012) the red line illustrates a plastic polymer, while the green line illustrates a highly elastic polymer. The highly elastic polymer, termed elastomer, is characterised by the high non-permanent deformation under low stress before failure.

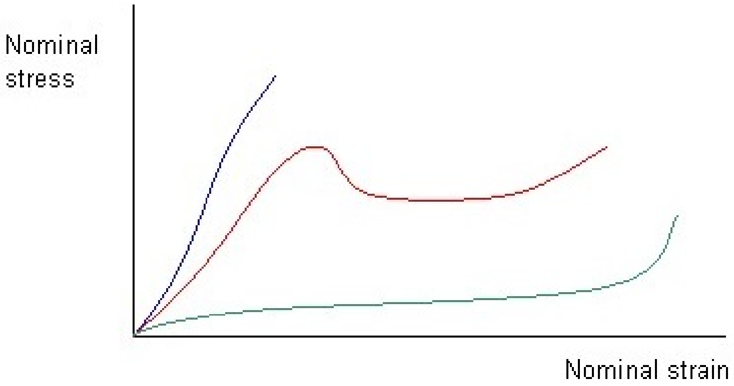

Figure 4.11: The Stress-Strain curve showing behaviour of different polymers (University of Cambridge 2015)

This increase in elasticity was noticed in Figures 4.2 to 4.3 and would be due to the increase in temperature. In Figure 4.3 the permanent deformation increased with temperature to 40°C. At 60°C the samples only underwent non-permanent deformation, which was illustrated in Figure 4.2. The compression testing of the 80°C samples, however, were continued to compressive failure. This explains why in Figure 4.3 the samples returned to originally shape like the 60°C samples but had fractured. The fracturing at one end, however could have been the result of the lumps noticed in Figure 4.1. Despite the fracturing, the samples had deformed elastically.

In Figure 4.4, the increase in elasticity also resulted in and change in the yield point. The yield point, where the material changes from elastic deformation to plastic deformation, is classified as the maximum on the curve for polymers (Callister & Rethwisch 2012). Corresponding with the peak stress, the increase in temperature reduced in a decrease in those values. The 60°C samples, did not have a yield point, confirming that the sample only deformed elastically in Figures 4.2 and 4.3. The 80°C samples followed a similar trend, although, did undergo compressive failure as shown by the slight curve maximum towards the end. This peak would have been the result of the fracturing, with the samples only deforming elastically despite that.

Callister & Rethwisch (2012) confirms that increasing temperature reduces Youngs Modulus, decreases strength and increases elasticity. This explains the observed physical trends and the decreasing trends of peak stress and youngs modulus in Table 4.1. With the change in values between 40°C and 60°C, in Figure 4.4 and Table 4.1, it would suggest that the glass transition temperature would be within that range.

The other trend noticed was the effect the white lumps had on the mechanical properties of the samples. Looking at the peak stresses of each 100% epoxy compression test, located in Appendix C, it was clear that the values varied. With the observation of the white lumps and their varying quantities in the samples, it can be assumed that they contributed to the variations in peak stress. It is also assumed that the lumps would have had an effect on the Youngs modulus. In Figure 4.1 the lumps had settled to one end of the samples and it was this end that failed in compression testing. Therefore the lumps, creating a plane of weakness in the samples, increased the ease of deformation to reduce the Youngs modulus.

With the lumps present in all samples it was assumed that all samples were equally effected, with decreased values. This allowed the samples to be compared and a trend analysis would eliminate most variations with lump quantities. Due to the minor variations with lump quantities, the increase of Youngs Modulus for the 30°C sample is considered irrelevant.

The white lumps would not have any direct effect on the glass transition range. This is due to the glass transition range being a property of the polymer when it changes from a solid to a rubber-like state (Michels et al. 2015). Therefore, the already decreased values, would still decrease further with the increase of temperature.

Specimen 80:20

With the increase in temperature a couple of trends were noticed for the 80:20 specimens. Firstly it was the behaviour of the stress-strain relationship curve with increasing temperature. It was mentioned in Section 4.2.2 that the relationship curves were same for the room temperature and 30°C 100:0 specimens. Therefore they followed reacted as a plastic polymer (Callister & Rethwisch 2012). This similarity can also be seen when comparing Figures 4.3 and 4.5. The samples had permanent compression after the compression testing, which increased with temperature.

For the specimens at 60°C and 80°C the trend followed the elastic polymer characteristics with high deformation over low stress. The difference between the 80:20 and 100:0 specimens was that the former had a clear yield point and there was less non-permanent deformation before the compressive failure. When comparing Figures 4.3 and 4.5 the samples showed minimal permanent compression. The compressive failure, however, resulted in greater fracturing with the bulging of the samples. Despite this, there was no degradation at one end of the sample due to a combination of the white lumps and temperature. Without the lumps it is assumed that the fracturing would be more uniformly spread across the surface. This uniform compressive failure is more ideal than the degradation of the 100% epoxy specimens. These improved mechanical properties suggest that the filler decreases the effect of the temperature on the polymer resin.

Lastly the 40°C specimens did not follow either the plastic or elastic polymer trend. This was due to there being large deformation under high stress. This different trend was also noticed in Table 4.2 as the sudden drop in mechanical properties occurred at different temperatures. For the Peak stress this was between 40°C and 60°C, while, for Youngs modulus it was between 30°C and 40°C. When comparing Figures 4.3 and 4.5 though, the 80:20 samples showed identical permanent deformation with the 100:0 samples. Based off the figures it was assumed that the glass transition temperature would be within the 40°C to 60°C range. With this assumption it suggest that there is inaccuracy of using the Youngs modulus to determine the effects of the temperature.

The white lumps present in the 100:0 specimens was assumed to be in the 80:20 specimens based off the similar failure ends and sticky residue on the samples. This suggest that the lumps were present in the mixture of the samples. It was also assumed that the white lumps had the same effect on the specimens irrespective of filler percentage.

Specimen 60:40

With the increase in temperature a couple of trends were noticed for the 60:40 specimens. Firstly it was the behaviour of the stress-strain relationship curve with increasing temperature. Comparing Figure 4.6 to 4.8 the relationship curves were similar. For the two lowest temperatures the 60:40 specimens had a more gradual peak stress and the peak stress had a reasonable drop with temperature. Despite these differences the curves were similar as they reacted as a plastic polymer (Callister & Rethwisch 2012).

Comparing the figures, the two highest temperatures had the same trend as they reacted as an elastic polymer (Callister & Rethwisch 2012). The only difference was that there was greater deformation before compressive failure and it sustained a greater peak stress. For the 40°C specimens they followed the trend in Figure 4.4. Despite this, the peak stress was not as large and it extended across a large deformation. Due to the yield point being spread across a large deformation, it suggests that the material acted elastically. Looking at Table 4.3, with the change in values between 40°C and 60°C, it suggests that the material was instead acting plastically.

Despite the similarities between the stress-strain relationship curves, there were no similarities when comparing Figures 4.3 and 4.5 to Figure 4.7. For the two former figures the compressive failure was either permanent compression or fracturing of the samples. In Figure 4.7 the compressive failure for all the specimens was shear failure, which was more evenly distributed at lower temperatures. It was decided that the white lumps were present in the specimens due to there being a sticky residue after the compressive failure. From this it was assumed that with increased filler and the higher density of the mixture, the white lumps would not have settled to bottom. This resulted in a higher consistency, explaining why the failure was evenly distributed along the specimen not restricted to one end. This suggest that the filler decreases the effect of the temperature and inconsistencies within the mixture on the polymer resin.

Specimen 40:60

With the increase in temperature a couple of trends were noticed for the 40:60 specimens. Firstly it was the behaviour of the stress-strain relationship curve with increasing temperature. Comparing Figure 4.8 to 4.10 the relationship curves were similar. For the two lowest temperatures the 40:60 specimens had minimal difference in peak stress with the increase in temperature. This is noticeable in Table 4.4 as the 30°C specimens had a higher average peak stress compared to the specimens at room temperature. Despite these differences the curves were similar as they reacted as a plastic polymer (Callister & Rethwisch 2012).

Comparing the figures, the two highest temperatures had the same trend as they reacted as an elastic polymer (Callister & Rethwisch 2012). The only difference was that there was less deformation before compressive failure and it sustained a greater peak stress. For the 40°C specimens the major difference was that the peak stress was no extended across a large deformation.

Comparing Figure 4.7 to 4.9, the compressive failure of shear cracking is similar. The biggest difference is that the samples were more brittle and the shear cracking was not evenly distributed across the surface. It is assumed that with less than 50% epoxy resin there was not enough adhesion of the filler to maintain the elasticity of the specimens. This contributed to the compressive failure at all temperatures being brittle and fracturing. It was mentioned in Section 4.2.4 that there was also voids present in the cured samples. The voids provide a plane of weakness for failure to occur, therefore, it explains why none of the shear failure was evenly distributed across the surface. These voids would have also contributed to the rough curves in Figure 4.10 as when the shear failure reached a void the deformation and stress would suddenly change.

4.3.2 Effects of Temperature

This section analyses the effect of temperature on each individual parameter.

Physical Observations

Figure 4.12 to 4.16 illustrates the comparison between the compression tested samples at each temperatures for each epoxy resin to filler ratio.

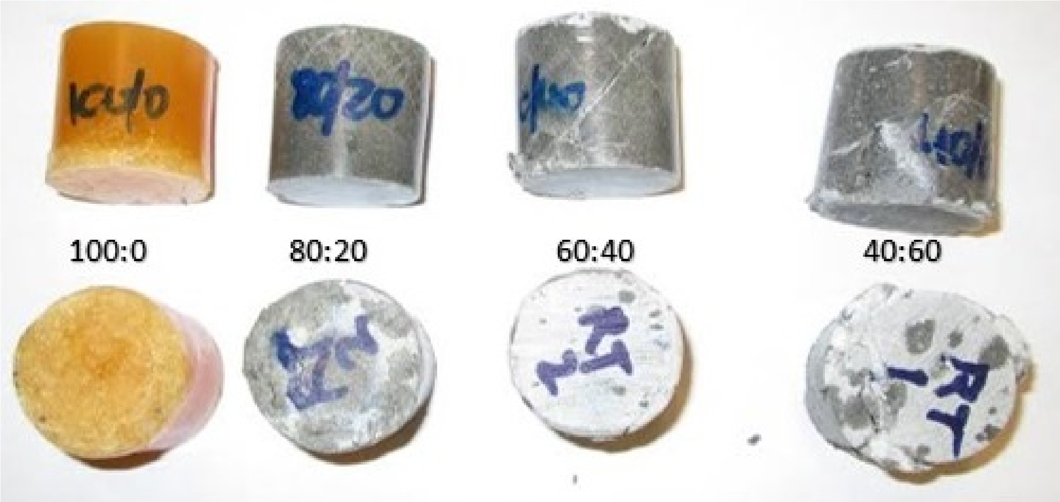

Figure 4.12: Room Temperature Compression Samples of Increasing Light-Weight Particulate Filler Percentage

Figure 4.12 shows the compression tested sample at room temperature at increasing light weight filler percentage. The figure illustrates that as the filler percentage increased the shear compressive failure of the samples was more evenly distributed. Shear failure also become more pronounced with the decrease in elasticity of the sample due to increasing filler.

The confinement of the compressive failure noticed in 0-20% filler, could be associated to the white lumps noticed in the samples. With the increase in filler content not only was the shear failure more evenly distributed, but the sticky residue noticed at the base of the samples decreased.

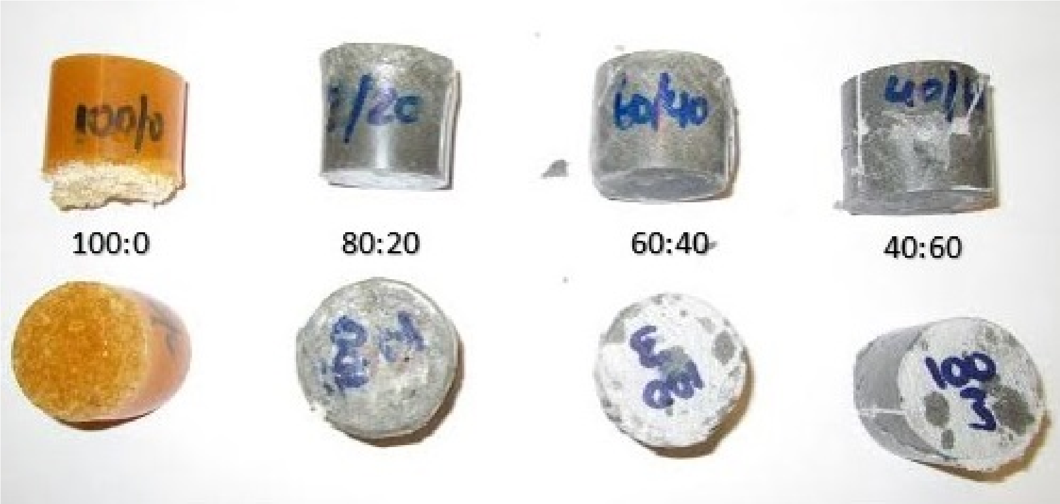

Figure 4.13: 30°C Compression Samples of Increasing Light-Weight Particulate Filler Percentage

Figure 4.13 shows the compression tested sample at 30°C at increasing light weight filler percentage. From Figure 4.13 the same trends noticed in Figure 4.12 were noted. These trends were that as the filler percentage increased the shear compressive failure of the samples was more evenly distributed and shear failure also become more pronounced.

There was also confinement of the compressive failure noticed in 0-20% filler samples and with the increase in filler content the sticky residue noticed at the base of the samples decreased.

Figure 4.14: 40°C Compression Samples of Increasing Light-Weight Particulate Filler Percentage

Figure 4.14 shows the compression tested sample at 40°C at increasing light weight filler percentage. From Figure 4.14 the same trends noticed in Figure 4.12 and 4.13 were noted. These trends were that as the filler percentage increased the shear compressive failure of the samples was more evenly distributed and shear failure also become more pronounced. There was also confinement of the compressive failure noticed in 0-20% filler samples and with the increase in filler content the sticky residue noticed at the base of the samples decreased.

With the increased temperature the samples at the higher filler percentage had minimal changes in permanent deformation when compared to Figure 4.13. At the lower filler percentages, shear compressive failure there was greater permanent compression and minimal shear failure crack were noted. The decrease in modulus of elasticity and the high elasticity of the epoxy resin has resulted in increased pliability, which decreased shear failure.

Figure 4.15: 60°C Compression Samples of Increasing Light-Weight Particulate Filler Percentage

Figure 4.15 shows the compression tested sample at 60°C at increasing light weight filler percentage. The trend in Figure 4.15 is similar to the trends noticed at lower temperatures which was that as the filler percentage increased the shear compressive failure of the samples was more evenly distributed. Shear failure, however, wasnt more pronounced as the most dramatic failure occurred at 80:20 epoxy resin to filler ratio. With the decrease in the modulus of elasticity across all samples it has resulted in increased pliability, which decreased shear failure.

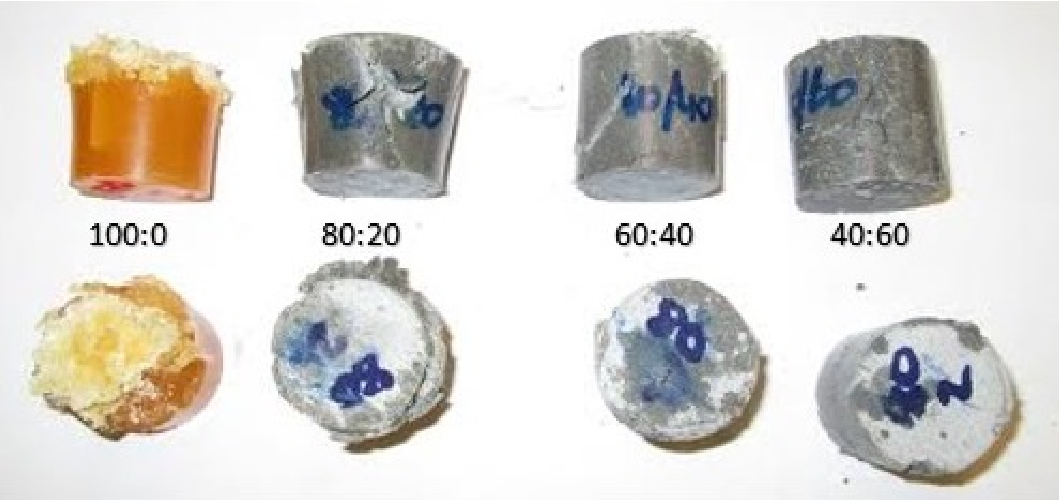

Figure 4.16: 80°C Compression Samples of Increasing Light-Weight Particulate Filler Percentage

Figure 4.16 shows the compression tested sample at 80°C at increasing light weight filler percentage. Unlike the compressive failure at the lower temperature the failure at 80°C resulted in more severe fracturing and cracking. It is also noted that the severity of the failure decreased with the increase of filler percentage. It was mentioned in Section 4.3.1 that the compressive failure of the 100% epoxy specimens had reached an elastic compressive failure (Callister & Rethwisch 2012). Meaning that the specimens had only deformed elastically before compressive failure. This would explain why with the decrease of epoxy resin the specimens had less severe compressive failure.

With the increased temperature the samples at the higher filler percentage had minimal changes in permanent deformation and still presented signs of shear failure. At the lower filler percentages, fracturing of the epoxy resin occurred before shear compressive failure resulting in greater permanent compression failure crack.

From the physical observations the specimens with a higher percentage of filler had less severe compressive failure at higher temperatures. This was because the filler reduced the percentage of the specimen that was effected the glass transition range. The filler also increased the density, and thus consistency, of the specimens providing a more uniform strength. The 40:60 specimens, however, did not contain enough epoxy to limit voids and crumbling upon failure. Therefore those specimens would not be recommended to be used. From the physical observations the 60:40 specimens were best.

Compression Testing

The two values that were determined from the compression testing were peak stress and Youngs modulus. Figure 4.17 to 4.18 illustrate the trend analysis of those parameters respectively. A trend analysis eliminates variations between samples and can be compared to determine the optimum mixing ratio.

The optimum mixing ratio of epoxy resin to light weight filler is the ratio that is least effected by the increase in temperature. This entitles the samples to have no significant reduction of the compressive strength and Youngs Modulus with the increase in temperature. From the trend analysis graph this is interpreted as the ratio trend with the straightest and least steep trend line.

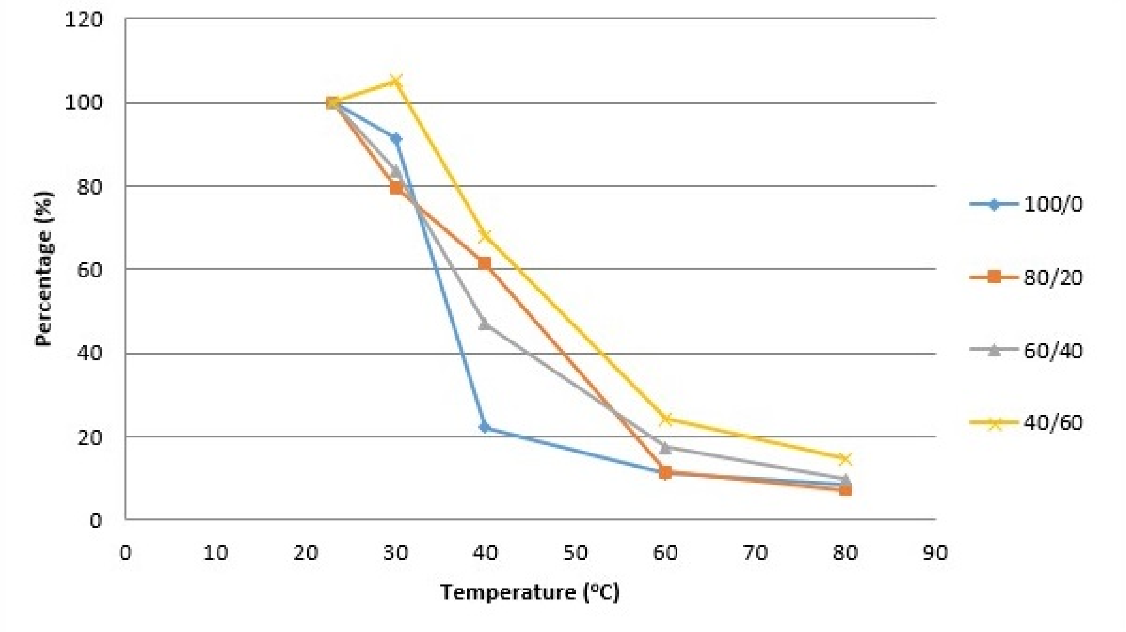

Figure 4.17: Peak Stress Trend Analysis for Resin/Filler Ratio

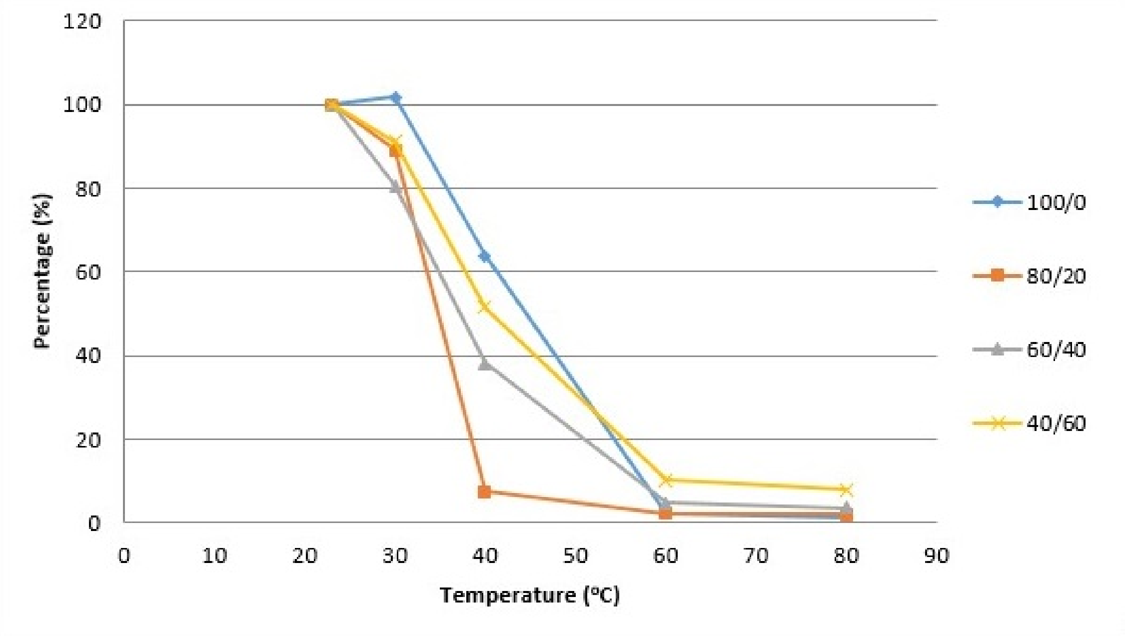

Figure 4.18: Young's Modulus Trend Analysis for Resin/Filler Ratio

From Figure 4.17 and Figure 4.18 the resin to filler ratio that was least effected by the increase in temperature was 40:60 epoxy resin to filler ratio. With an overall decrease of 85.2% and 92.1% in peak stress and Youngs modulus respectively, this suggests that the 40:60 specimens had the optimum mixing ratio. Despite this, the 40:60 was almost unworkable and resulted in voids within the sample. The voids disrupt the consistency of the sample and provide a plan of weakness for shear and brittle failure. The voids also result in the specimens being permeable, this counteracts the purpose of the epoxy resin being used for its impermeability to cover materials (Lokuge & Aravinthan 2013, Muthukumar & Mohan 2004). For these reasons the 40:60 ratio was disregarded as the optimum mixing ratio.

From Figure 4.17 and Figure 4.18 the second best mixing ratio was the 60:40 epoxy resin to filler ratio. Between room temperature and 80°C there was a decrease of peak stress and Youngs modulus of 90.2% and 96.4% respectively. When comparing the compressive failure to the 40:60 ratio in Figures 4.12-4.16, the shear failure was also evenly distributed over the sample. With the increased elasticity of the samples the compressive failure was less brittle and severe than the 40:60 ratio. In sample preparation the mixture was workable and with consistency similar to concrete voids could be prevented using conventional method. Therefore it was concluded that the optimum mixing ratio is 60:40.

When looking at the compression testing results in Section 4.2 a couple of trends were noticed. From the stress-strain relationship curves, at 60°C and above the specimens underwent elastic deformation instead of a plastic deformation (Callister & Rethwisch 2012). This suggests that, despite the addition of filler, the specimens had a glass transition temperature between 40°C to 60°C. Ferdous et al. (2015) confirms this from DMA analysis of the same commercially available materials used in this study.

Differential Thermal Analysis (DTA) is one of the commonly used testing methods used for thermal analysis of a material. The other methods include the Differential Scanning Calorimetry (DSC), Thermal Gravimetric Analysis (TGA), and Thermo Mechanical Analysis (TMA) (Jin & Park 2012, Michels et al. 2015, Park et al. 2005, Zhou & Lucas 1999).

Figure 4.19 illustrates the results of the DMA analysis performed by Ferdous et al. (2015). From the figure it can be noticed that despite the increase in filler the glass transition range of 50°C to 60°C did not change. This is to be expected as the filler does not change the composition of the epoxy matrix. The filler did, however, decrease the drop in storage modulus with the increase of temperature. Jin & Park (2012) confirms this as with the addition of filler in his experiments there was a decrease in the drop of storage modulus. While, in Study 1 the Youngs modulus was determined the trend aligns with the results in Figure 4.18.

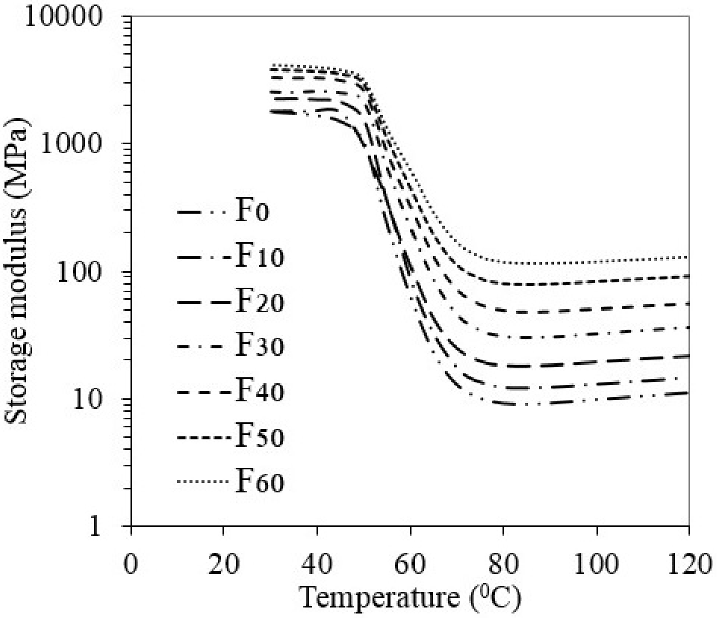

Figure 4.19: Storage Modulus vs Temperature (Ferdous et al. 2015)

The glass transition range of 50°C to 60°C could be compared to similar ranges determined by Moreira, Sphaier, Reis & Nunes (2012), Michels et al. (2015) and Elalaoui et al. (2012). Determining the actual glass transition range, however, was outside the scope of this report. The main aim is to investigate the effects of elevated temperature on mechanical properties, which was determine by analysing the compressive behaviour. The equipment used to determine the glass transition temperature cannot be used to measure the mechanical properties. Therefore, an analysis of the glass transition temperature is not required.

Due to the low region of the glass transition temperature, though, Michels et al. (2015) encouraged post-curing where possible as it significantly increases the glass transition temperature. Michels et al. (2015) also specified the importance of determining the maximum service temperature the structure can be exposed to. Table 4 within Glass transition evaluation of commercially available epoxy resins used for civil engineering applications (Michels et al. 2015) summarises several guidelines for the assessment of the glass transition temperature and allowed service temperature. Due to the inconsistency between the guidelines, in future unification would be required.

When comparing the Youngs modulus, Moreira et al. (2012) stated that the epoxy sample cured at ambient temperatures, had a modulus of elasticity between 2.4-5.0 GPa. All the experimental values of the epoxy sample was outside this range, with the highest value recorded for the 40:60 PFR ratio at 1.37 GPa in Table 4.4. Despite this result suggesting that the epoxy sample was not cured optimally, Moreira obtained the results from flexural and tensile testing. Materials arent expected to perform similarly in tension and compression, therefore the values are still valid.

Comparing the Tables 4.1 to 4.4 it was noticed that at lower temperatures the 80:20 specimens had the highest peak stress. The peak stress then decreased with additional filler. From the results it is unknown whether this is due to there being less matrix to wet the filler or the filler having a lower strength. In Figures 4.12 to 4.16 with the crumbling of the 40:60 specimens upon failure, it suggest that there was no enough epoxy to wet the filler. This was not noticed in the 60:40 specimens as the failure was instead distributed evenly without crumbling of the specimens. It is, however, unknown how the white lumps effected the peak stress in the specimens. Therefore, further experimentation would be required for more accurate results.

4.3.3 Limitations and Problems

Limitations

Majority of the limitations for Study 1 reflected the practicality of the results for commercial use. In Chapter 2 the literature stated that post cured samples had increased mechanical properties, including an increased glass transition temperature (Custdio et al. 2011). Ideally the epoxy samples would be post cured, however, for economical and practical reasons not all epoxy applications can be post cured. Therefore the study limited the curing of the samples to ambient temperatures. For practical and economic reasons the epoxy resin used was also limited to a commonly used commercially available product.

Another limitations for Study 1 was the equipment available for testing. It was mentioned in Section 4.3.2 that DMA analysis was outside the scope of the report as the exact glass transition temperature wasnt required to determine the optimum mixing ratio. The optimum mixing ratio of epoxy resin to light weight filler is the ratio that its mechanical properties are least effected by the increase in temperature. While the glass transition temperature does effect the mechanical properties of the epoxy, it is not a defined material property due to varying with curing and testing parameters (Michels et al. 2015). This information is often excluded from commercial product data (Custdio et al. 2011). Requiring to experimentally access that information reduces the practically of that result in this report being commercially used. The DMA also did not measure the compressive behaviour, therefore, no equipment was used for thermal analysis of the specimens.

For the compression testing the testing equipment was limited to a 100 kN capacity compression testing machine. This machine was used due to its accessibility at the University of Southern Queensland. It was mentioned in Section 4.2.1 that the Youngs modulus values were calculated from based on the calculated strain from the displacement of the machine. In Tables 4.1 to 4.4 the values at 80°C seem low. To further verify this values it is recommended that strain gauges are attached to specimens in future testing.

Instead of the glass transition temperature the mechanical properties of the specimen, including the peak stress and Youngs modulus, were measured. Determining the rate of reduction for these two properties allowed the optimum mix to be determined. Measuring the Youngs modulus of the sample to illustrate its dependence on temperature, has been performed by Neitzel et al. (2012) and the graphical results are presented in Figure 4.20. From this figure it suggests that Youngs modulus can be used to determine the effect of temperature. In this figure it clearly shows that even with the addition of filler the glass transition range can be determined by the increased rate of decline in the modulus of elasticity. This can be compared to Figure 4.21, which is a similar graph based on the results for the different mixes and temperature. From the comparison an increased rate of decline can be seen with the increase of temperature.

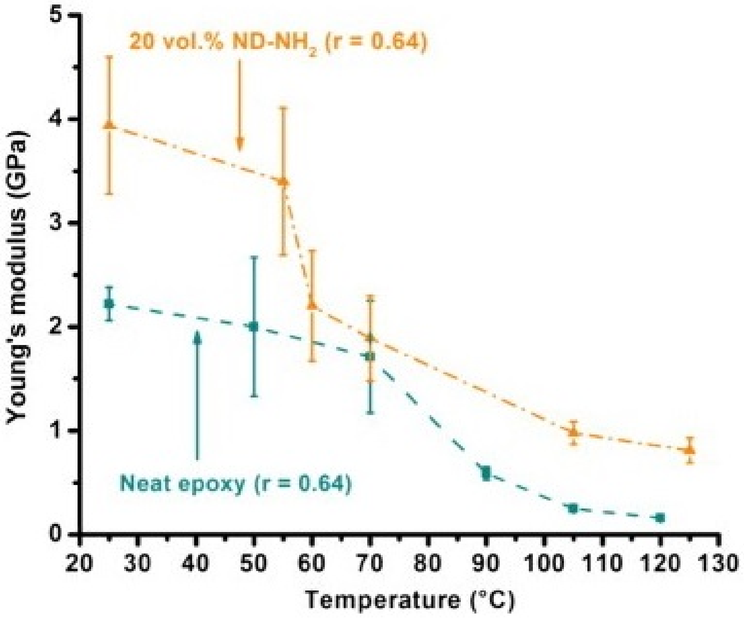

Figure 4.20: Dependence of Young's Modulus on Temperature (Neitzel et al. 2012)

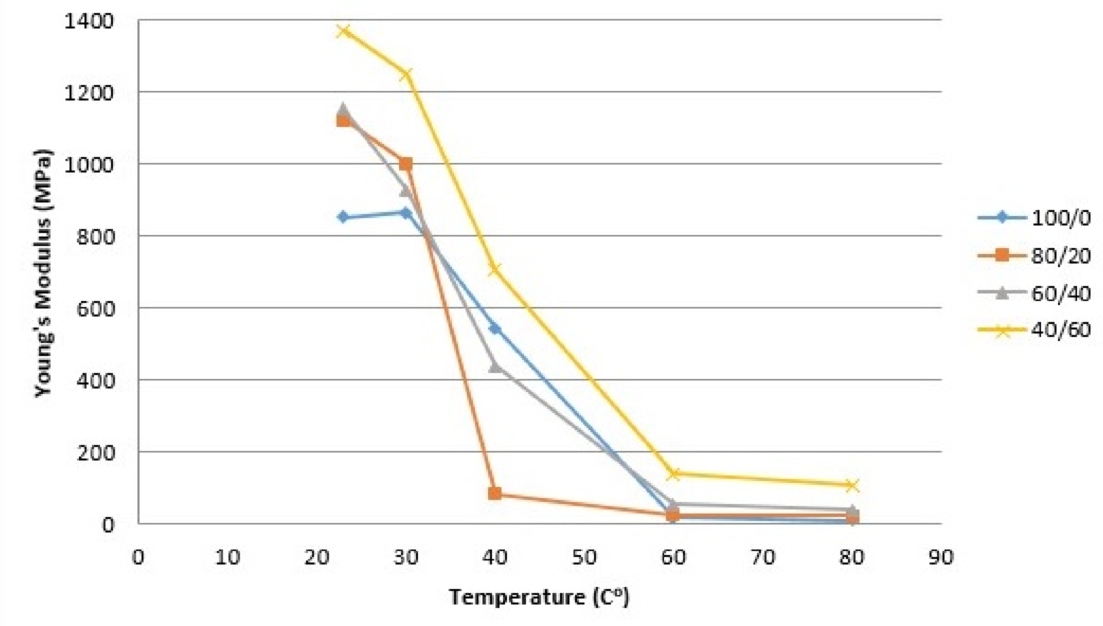

Figure 4.21: Change of Young's Modulus with Temperature

The only problem with using the Youngs Modulus to determine the glass transition range was finding literature to compare to. Most of the literature contained in Chapter 2 used either DSC, TGA, DTA, or TMA to determine an accurate value for glass transition temperature. In these methods the effect of temperature is measured in terms of weight (%), heat flow, tan delta or storage modulus (Jin & Park 2012). None of these terms are determine from the current testing procedure for Study 1. Sirk, Khare, Karim, Lenhart, Andzelm, McKenna & Khare (2013), however, concluded that the while the Youngs modulus was not the same as the storage modulus for the glassy and rubbery plateau regions of the sample they exhibited a close numerical agreement. Therefore the glass transition range calculated from the storage modulus can be compared to the experimental results from Study 1.

Problems

In Study 1, two problems were encountered that would have altered the experimental results. The first was mentioned in Section 4.2.1, which was tested the samples at 30°C instead of the originally planned 100°C. Due to the testing taking place after the 80°C samples, two of the 100% epoxy samples couldnt be used in the comparison.

The second problem was in relation to the white lumps noticed at the bottom of the 100% epoxy sample. Whilst the light-weight particulate filler resulted in the higher filler percentage samples to be opaque, it was assumed that the lumps were additionally present. This was based off similar observations during the compression testing. All showed signs of sticky residue after compression and the 80:20 epoxy resin to filler ratio clearly had a plane of weakness towards one end.

Despite the plane of weakness being less noticeable in the 60:40 and 40:60 epoxy resin to filler ratios, it was assumed the white lumps were still present. Signs of sticky residue, though minimal in comparison, were still occurring. It was hypothesized that the light weight filler increased the density of the samples, resulting in the white lumps not settling to the bottom of the sample during curing. This assumption was supported by the filler increasing the stiffness and decreasing the workability of the samples.

Based off the assumption that the white lumps were present in all the samples it was concluded that they were lumps of solidified Part A. This conclusion was based off an observation during the sample preparation. When opening the container of Part A there was a top layer of solidified material. Underneath this layer the Part A was at a normal consistency. Despite trying to break up and mix in the solidified layer of Part A, there were still lumps present in mixture. Even in the preparation of the samples the lumps didnt disappear.

These lumps still contributed to the mixing weights of the ingredients in the sample preparations. Due to the lumps not properly mixing with the amine hardener (Part B) there would have been excess Part B in the mixture upon curing. This would explain the sticky residue that was secreted from the samples upon compression. If the filler material absorbed some of the Part B it would also explain why there was less sticky residue with the increase of filler percentage.

Due to the solidification of Part A, it wouldnt have mixed with any of the other materials. Therefore it most likely only reduced the consistency of the sample, acting similarly to air voids. It would have resulted it weak spots in the epoxy matrix and would have reduced the peak stress of the samples. It is unknown the full extent the excess Part B would have had on the mechanical properties of the samples. The most plausible explanation would be that it resulted in voids in the epoxy matrix, like Part A. Due to its fluidity over Part A though, it provided no mechanical strength as it easily seeped out. This would have resulted in changes to the peak stress and modulus of elasticity.

Despite the variation in Part B seepage between the filler percentages the results in Study 1 would still be valid. The trend analysis would eliminate any variations and the results would reflect the effect of temperature.

4.4 Summary

Chapter 4 determined the optimal filler content, with respect to elevated temperature, that had the least reduction in compressive strength of the epoxy resin. The 40:60 specimens were the least effected by the increase in temperature, with an overall decrease of 85.2% and 92.1% in peak stress and Youngs modulus respectively. Due to low workability, voids and brittle compressive failure though, it was decided that the specimen could not be used practically.

The 60:40 specimens were the second least effected from the increase in temperature, with an overall decrease of 90.2% and 96.4% in peak stress and Youngs modulus respectively. Minimal voids were present on the specimens and compressive failure was an evenly distributed shear failure. The density of the specimen also prevented the white lumps present in the mixture from settling to the bottom. Due to the unknown effect of the white lumps, it is recommended that they are removed from the mixture in Chapter 5.

From Chapter 4, it can be concluded that the optimal mix design was determined to be the 60:40. This is due to its having a minimal reduction of strength with temperature and not undergoing brittle compressive failure. The 60:40 mix will be used in Chapter 5, which tests the durability of the optimum mix.

Chapter 5

Effects of Simulated Environment on Durability

5.1 Introduction

This section presents the results and observations as well as the discussion on the durability of epoxy-based polymer matrix when exposed to simulated environments. In Chapter 4, it was concluded that the optimal epoxy based polymer matrix was 60% epoxy resin and 40% light weight particulate filler. In Chapter 3, the preparation of the sample was presented. These specimens were exposed to either air, water, salt-water up to 6 months or hygrothermal environments up to 1 month. The specimens were tested under compression at pre-set intervals, presented in Chapter 3. The physical observations, load-deformation relationship, dimensions, weight and microscopic observations of the specimens with different environmental exposure are presented. Analysis and discussion on the effects of the simulated environments on the parameters considered in this study are also presented.

5.2 Results and Observations

The collected data from the testing is provided in Appendix D. The results and observations are presented separated based on exposure to simulated environment. Analysis and discussion with respect to the effect of temperature is presented in Section 5.3.

5.2.1 Air Environment

Qualitative Results

Qualitative results include the physical observations and the microscopic observations. Physical observations were made to all specimens. The observations noticed for specimens exposed to the air environment are illustrated in this section and are separated into physical observations and microscopic observations.

PHYSICAL OBSERVATIONS

The initial testing were not categorised under any environment so that they could be used for comparison of all environments. Figure 5.1 and 5.2 illustrates the compression tested samples exposed to air over increasing duration. With the increase of duration the shear failure of the samples becomes more pronounced.

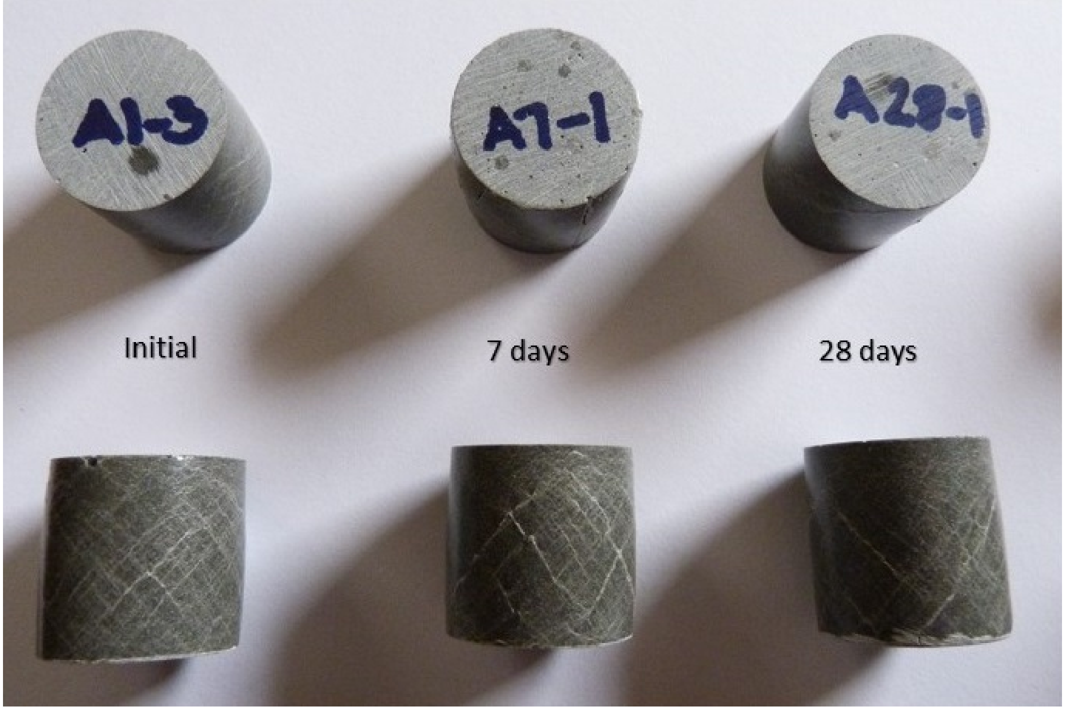

Figure 5.1: Air Environment Compression Samples at Increasing Duration (Initial to 28 Days)

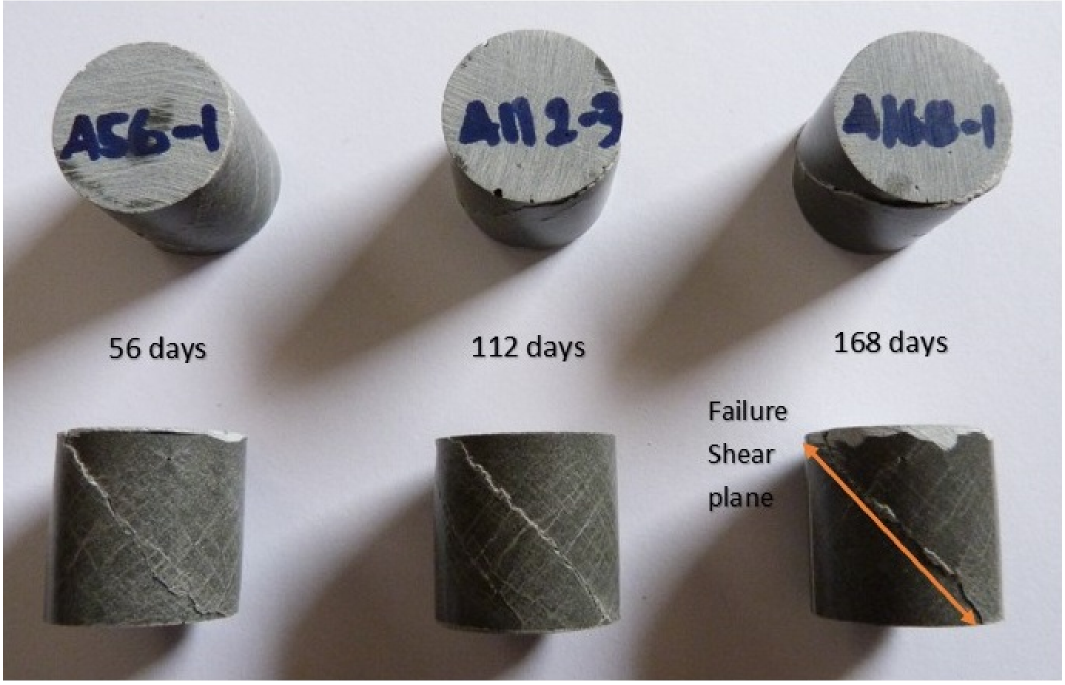

Figure 5.2: Air Environment Compression Samples at Increasing Duration (56 to 168 Days)

To emphasise how exposure to the air environment effected the compressive failure of the samples a close up comparison between the initial and 168 days is provided in Figure 5.3. After the 168 days the surface of the samples are unchanged. Compressive failure is, however, less distributed and more pronounced long one major shear plane.

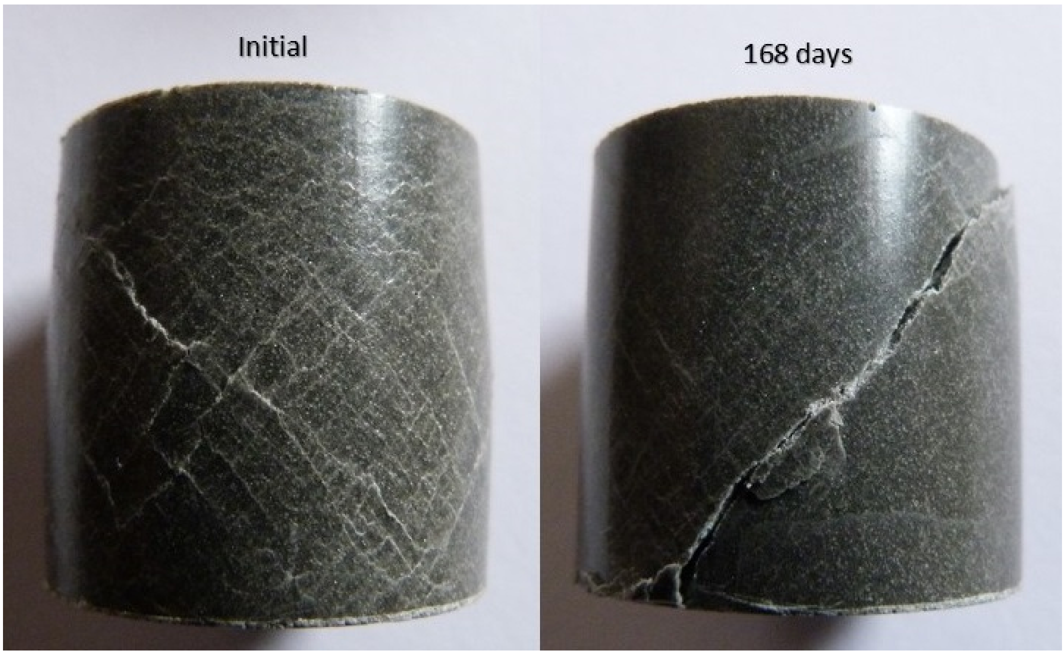

Figure 5.3: Comparison Between Initial and 168 Days Compression Testing for Air Environment

MICROSCOPIC OBSERVATIONS

The microscopic observations were originally planned to be taken for the whole duration of Study 2. When the microscopic observations of the 112 day samples were taken (Figure 5.5), they were compared to the initial sample (Figure 5.4). From the comparison there was no discernible difference between the images. This either suggests that a stronger magnification was required to determine changes in the epoxy matrix or that exposure to air resulted in no changes. Irrespective of the reason, microscopic observations werent performed for 168 days and 7 to 56 day results are provided in Appendix D.

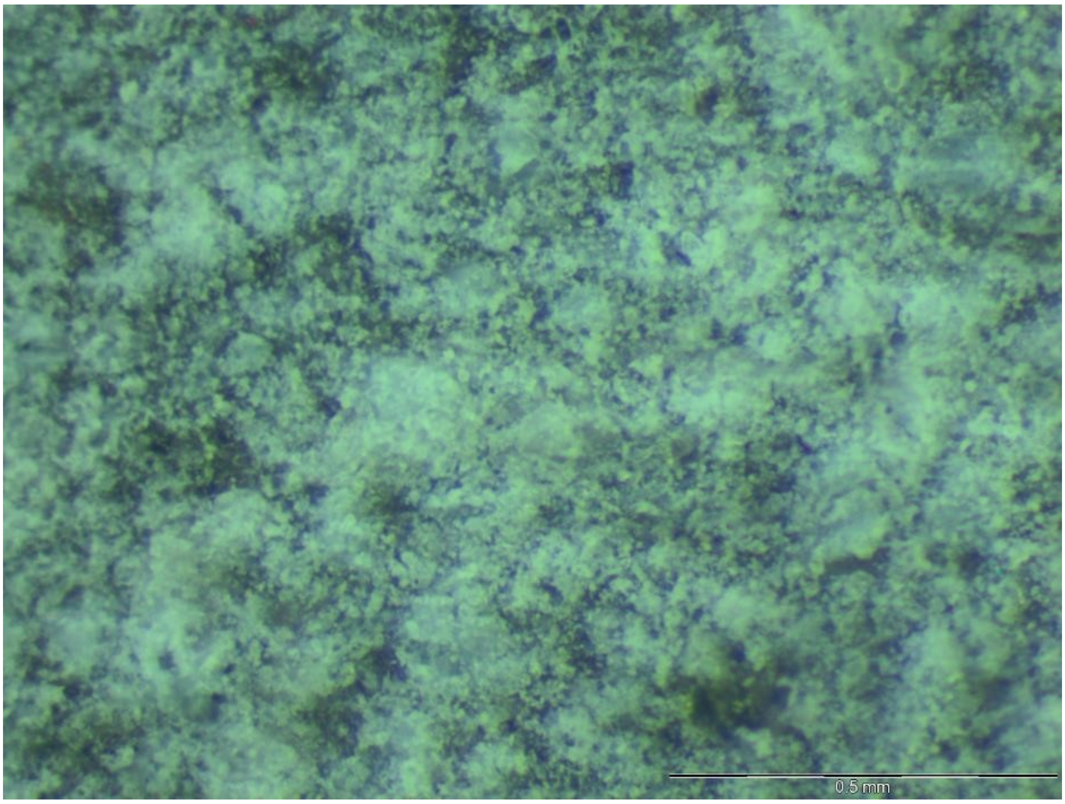

Figure 5.4: Microscopic Observation of Initial Sample

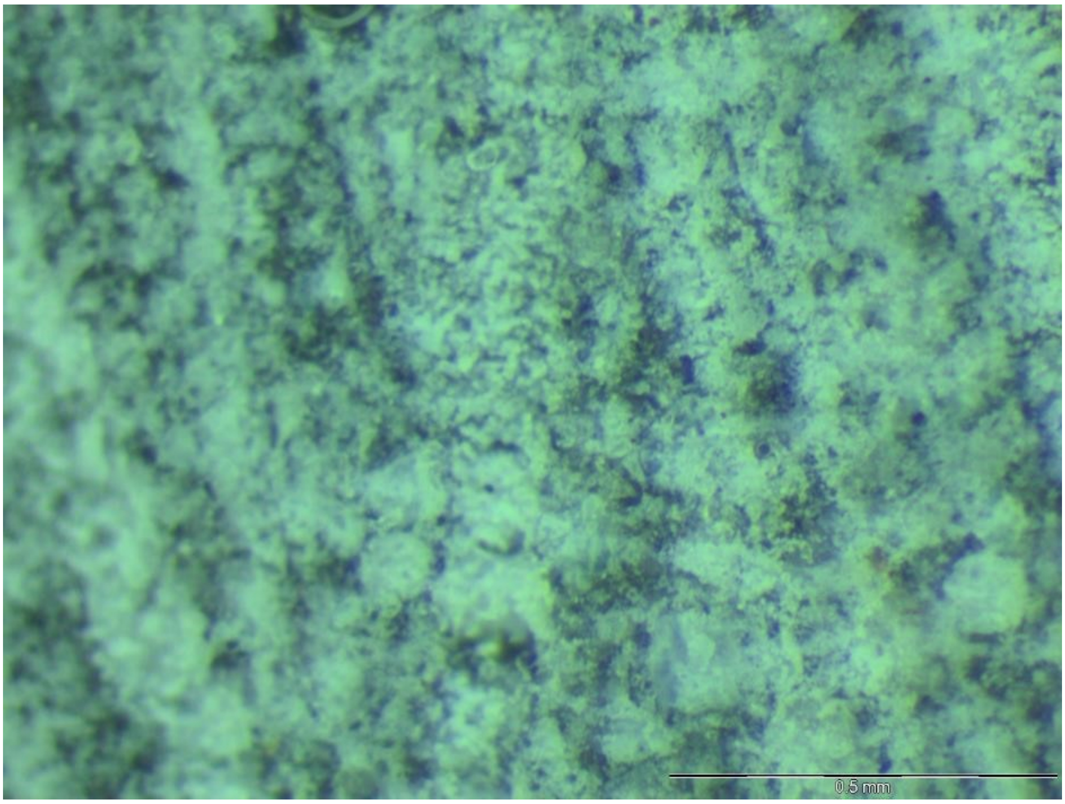

Figure 5.5: Microscopic Observation of 112 Day Sample Exposed to Air Environment

Quantitative Results

This section contains the quantitative results for the compression testing, dimension and weight changes (Table 5.1). Figure 5.6 shows the stress-strain behaviour under the compression testing on the specimens exposed to air. With the increase in duration the peak stress and modulus of elasticity appear to have increased, as proved in Table 5.1.

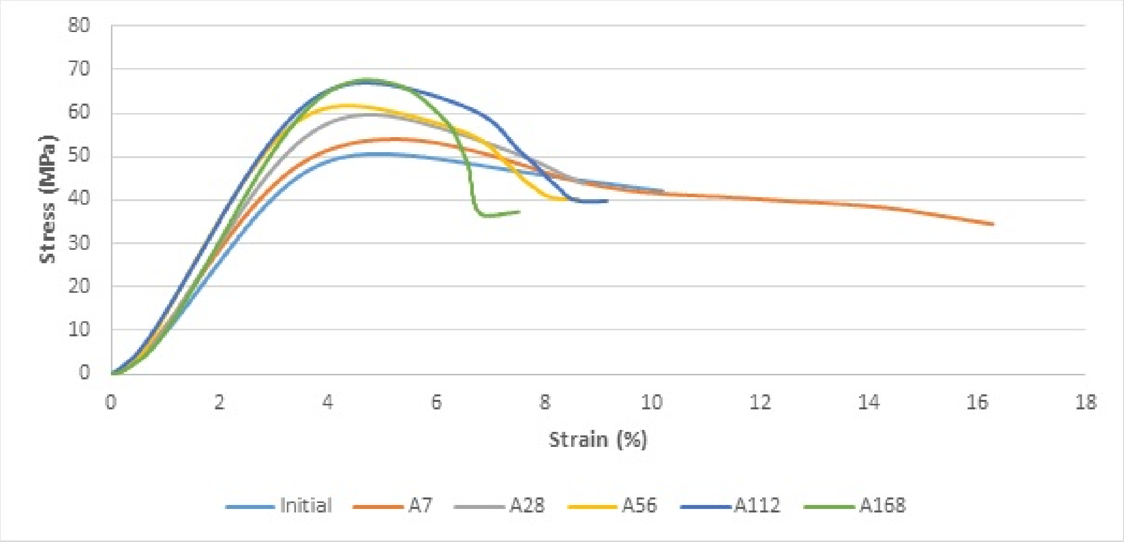

Figure 5.6: Stress vs Strain Graph for Specimens Exposed to Air Environment

In Table 5.1 the diameter, height and weight changes have be presented by trend analysis. From the initial measurements of all the specimens before environmental exposure, this shows the percentage changes of those parameters. Peak load, and peak stress, however, the exact average values were provided as the specimens can only be tested once. Youngs Modulus was determined by plotting only the slope of the stress vs strain graph in excel. A linear trend line was then applied and from the equation of the line the slope was converted to MPa. It was previously mentioned in Section 4.2.1 the data for the stress vs strain was chosen arbitrarily based on which better illustrated the stress vs strain trend

It should also be noted that the two bottom rows are represented as day 121 and 169 instead of 112 and 168 respectively. This was because testing was performed on those days instead of their preassigned days. For accuracy in graphing the results later on in the chapter, there are presented on their actual day of testing.

Table 5.1: Quantitative Results for Specimens Exposed to Air Environment

Day	**Diameter**	**Height**	**Weight**	**Peak Load kN**	**Peak Stress MPa**	**Young's Modulus MPa**
0	1	1	1	34.53	50.03	1573.7
1	1.00282	1.00175	1.00001	-	-	-
3	1.00158	1.00315	1.00002	-	-	-
7	0.99915	0.99720	1.00002	36.77	53.62	1616.4
28	1.00073	0.99688	1.00029	41.37	60.11	1877.6
56	1.00073	0.99783	1.00042	42.91	62.20	1982.2
121	1.00237	1.00777	1.01104	44.09	64.13	2039.9
169	1.00462	1.00415	1.00003	46.94	68.46	2123.0

From Table 5.1 there is no clear trend for duration and the parameters diameter, height and weight. With the increase of duration, though, there is also an increase in peak load, peak stress and youngs modulus.

5.2.2 Water Environment

Qualitative Results

PHYSICAL OBSERVATIONS

Figure 5.7 and 5.8 illustrates the compression tested samples exposed to water over increasing duration. With the increase of duration there is no clear trend as shear failure is more pronounced in some specimens but evenly distributed in others.

Figure 5.7: Water Environment Compression Samples at Increasing Duration (Initial to 28 Days)

Figure 5.8: Water Environment Compression Samples at Increasing Duration (56 to 168 Days)

To emphasise how exposure to the water environment effected the compressive failure of the samples a close up comparison between the initial and 168 days is provided in Figure 5.9. After the 168 day the surface of the samples appears to have increased number of large voids. The voids provide a plane of weakness, possibly resulting in less pronounced shear failure. Compressive failure is distributed similarly with shear failure, while more pronounced, appears to have taken a path of least resistance.

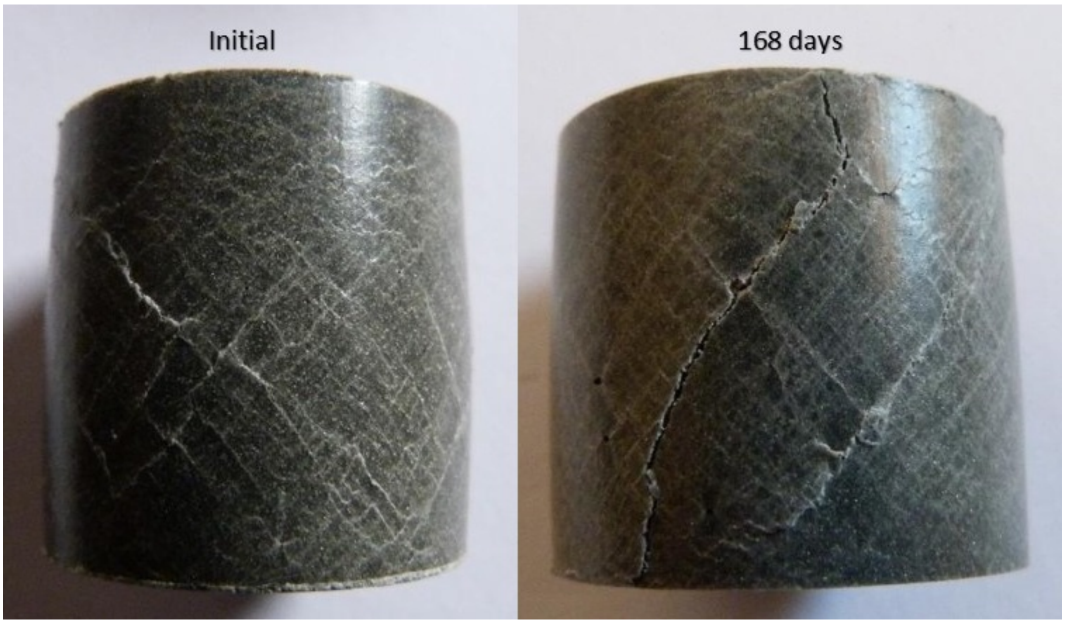

Figure 5.9: Comparison Between Initial and 168 Days Compression Testing for Water Environment

MICROSCOPIC OBSERVATIONS

The microscopic observations for specimens exposed to the water environment were also stopped after 112 days. When the microscopic observations of the 112 day samples were taken (Figure 5.10), they were compared to the initial sample (Figure 5.4). Similar to the air environment there was no clear differences between the images, again suggesting a stronger magnification was required. The 7 to 56 day results are provided in Appendix D.

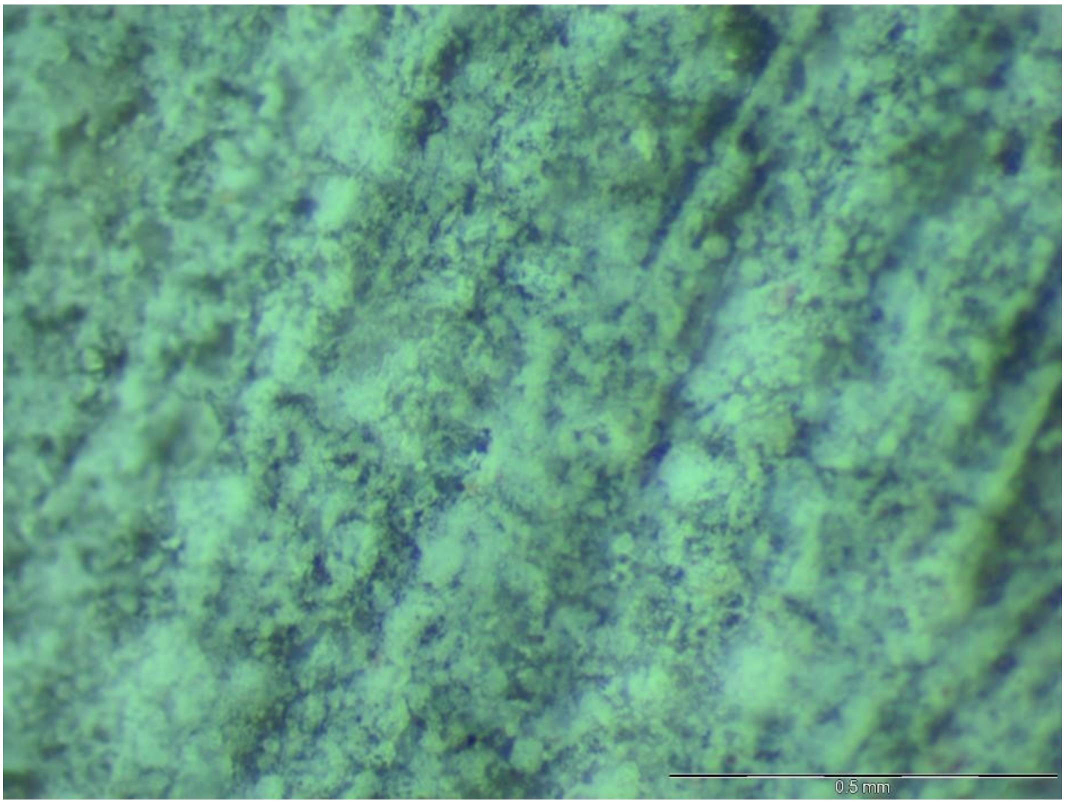

Figure 5.10: Microscopic Observation of 112 Day Sample Exposed to Water Environment

Quantitative Results

This section contains the quantitative results for the compression testing, dimension and weight changes (Table 5.2). Figure 5.11 shows the stress-strain behaviour under the compression testing on the specimens exposed to water. With the increase in duration the peak stress appear to have increased.

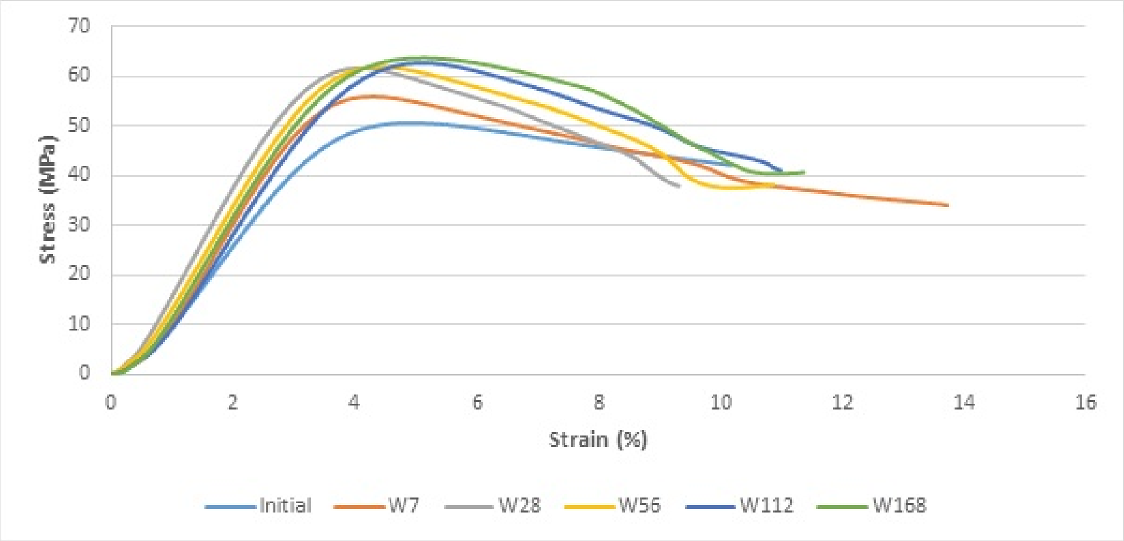

Figure 5.11: Stress vs Strain Graph for Specimens Exposed to Water Environment

From Table 5.2 there is no clear trend for duration and the parameters diameter, height and youngs modulus. With the increase of duration, though, there is an increase in peak load, peak stress and weight that either tapers or slightly declines at the 169 day mark.

Table 5.2: Quantitative Results for Specimens Exposed to Water Environment

Day	Diameter	Height	Weight	Peak Load kN	Peak Stress MPa	Young's Modulus MPa
0	1	1	1	34.53	50.03	1573.7
1	1.00135	0.99786	1.00063	-	-	-
3	1.00028	0.99211	1.00119	-	-	-
7	0.99943	0.99081	1.00148	37.85	53.70	1935.5
28	0.99938	0.99420	1.00204	41.09	60.15	2028.5
56	1.00017	0.99538	1.00282	41.72	60.70	2017.6
121	0.99887	0.98962	1.00368	42.58	62.17	1853.3
169	0.99797	0.98516	1.00334	42.44	62.17	1969.0

5.2.3 Salt-Water Environment

Qualitative Results

PHYSICAL OBSERVATIONS

Figure 5.12 and 5.13 illustrates the compression tested samples exposed to salt-water over increasing duration. With the increase of duration the shear failure of the samples becomes more pronounced. The 112 day sample also appears to have voids present on its surface.

Figure 5.12: Salt-Water Environment Compression Samples at Increasing Duration (Initial to 28 Days)

Figure 5.13: Salt-Water Environment Compression Samples at Increasing Duration (56 to 168 Days)

To emphasise how exposure to the salt-water environment affected the compressive failure of the samples a close up comparison between the initial and 168 days is provided in Figure 5.14. After the 168 day the surface of the samples appears to have increased number of voids, although the voids are not as large as the voids in the 112 day specimen in Figure 5.13. With the size of the voids relatively small the specimen was still had a pronounced shear failure as noted in Figure 5.13.

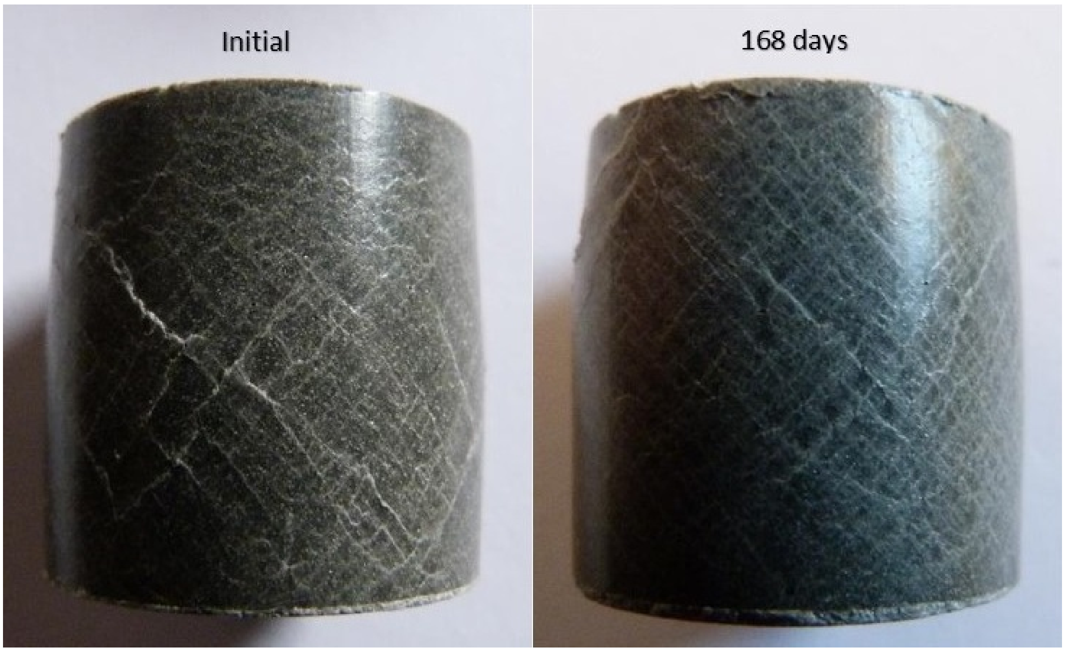

Figure 5.14: Comparison Between Initial and 168 Days Compression Testing for Salt-Water Environment

MICROSCOPIC OBSERVATIONS

The microscopic observations for specimens exposed to the salt-water environment were also stopped after 112 days. When the microscopic observations of the 112 day samples were taken (Figure 5.15), they were compared to the initial sample (Figure 5.4). Similar to the air and water environment there was no clear differences between the images, again suggesting a stronger magnification was required. The 7 to 56 day results are provided in Appendix D.

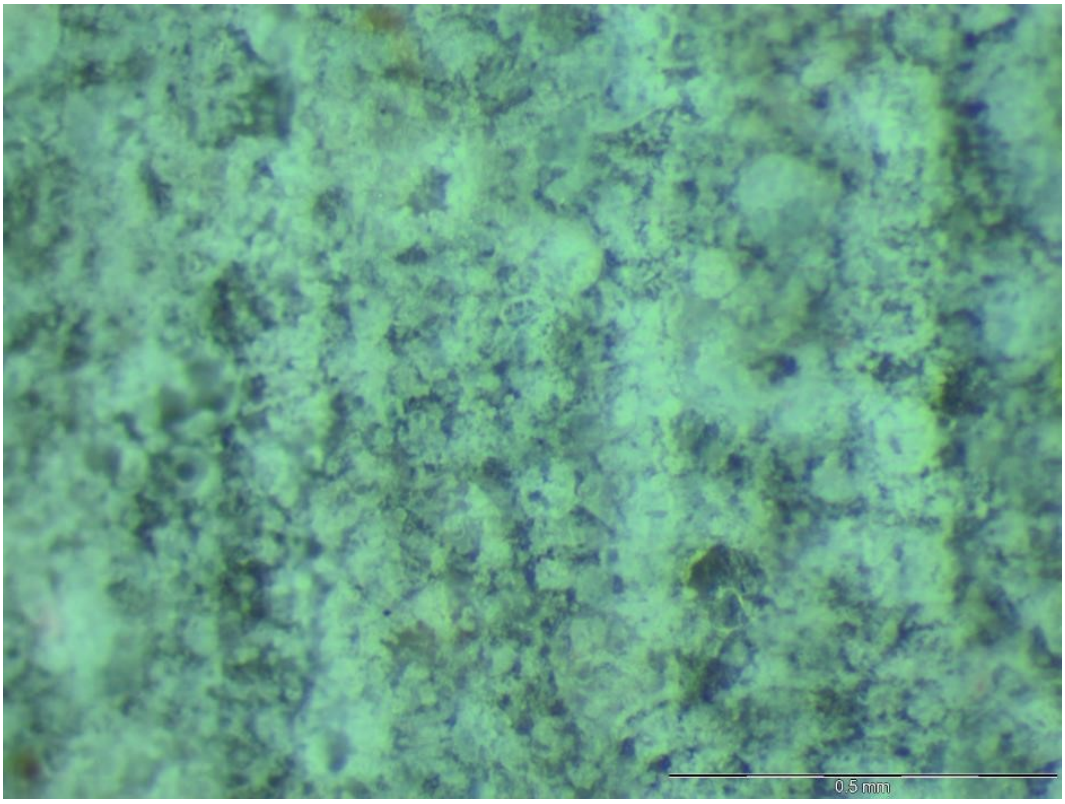

Figure 5.15: Microscopic Observation of 112 Day Sample Exposed to Salt-Water Environment

Quantitative Results

This section contains the quantitative results for the compression testing, dimension and weight changes (Table 5.3). Figure 5.16 shows the stress-strain behaviour under the compression testing on the specimens exposed to salt-water. With the increase in duration the peak stress and modulus of elasticity appear to have increased to 112 days then declined.

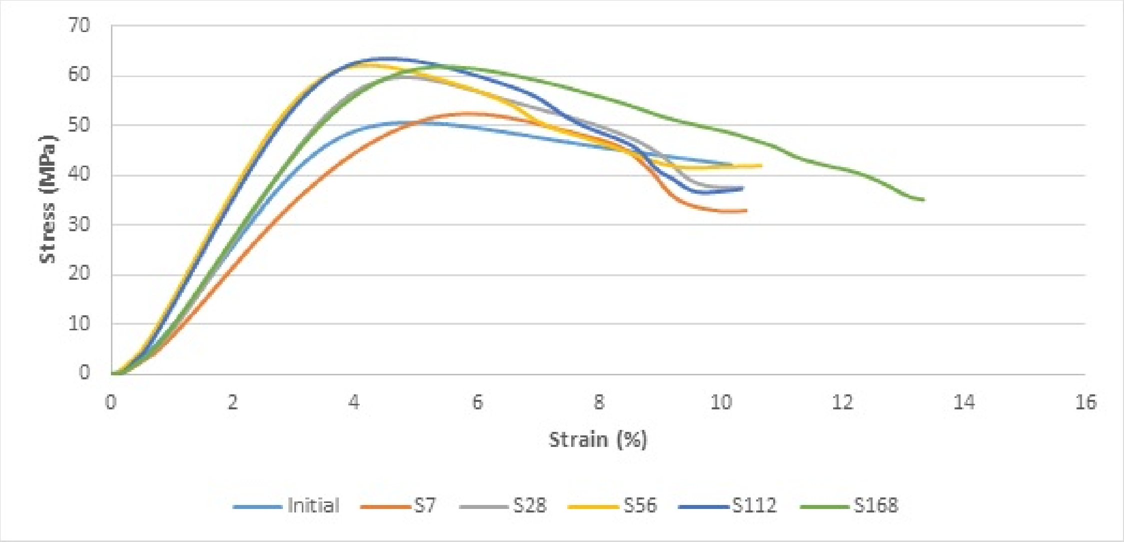

Figure 5.16: Stress vs Strain Graph for Specimens Exposed to Salt-Water Environment

From Table 5.3 there is no clear trend for duration and the parameters diameter and height. With the increase of duration, though, there is an increase in peak load, peak stress and youngs modulus to 56-121 days as noted in Figure 5.16. After that point the values started to decline. The weight of the samples increased for the whole duration of the experiment.

Table 5.3: Quantitative Results for Specimens Exposed to Salt-Water Environment

Day	**Diameter**	**Height**	**Weight**	**Peak Load kN**	**Peak Stress MPa**	**Young's Modulus MPa**
0	1	1	1	34.53	50.03	1573.7
1	1.00192	0.99798	1.00039	-	-	-
3	1.00192	0.99315	1.00104	-	-	-
7	0.99932	0.99174	1.00124	36.63	53.69	1356.1
28	1.00062	0.99389	1.00123	41.25	59.87	1804.0
56	0.99932	0.99114	1.00219	42.39	62.01	2113.3
121	0.99882	0.98505	1.00292	42.81	62.71	2065.6
169	0.99809	0.98206	1.00319	42.48	61.93	1731.6

5.2.4 Hygrothermal Environment

Qualitative Results

PHYSICAL OBSERVATIONS

Figure 5.17 and 5.18 illustrates the compression tested samples exposed to hygrothermal environment over increasing duration. With the increase of duration there is no clear trend as shear failure is more pronounced in some specimens but evenly distributed in others. The blue marker used to number the samples was partially absorbed by the water, resulting in the water turning blue. This was beneficial as it illustrated whether the specimens were absorbing the water by the blue staining. This can blue staining is greatly noticeable on all unsealed surfaces, including the sanded end and the cut from the hacksaw when removing the samples from their moulds.

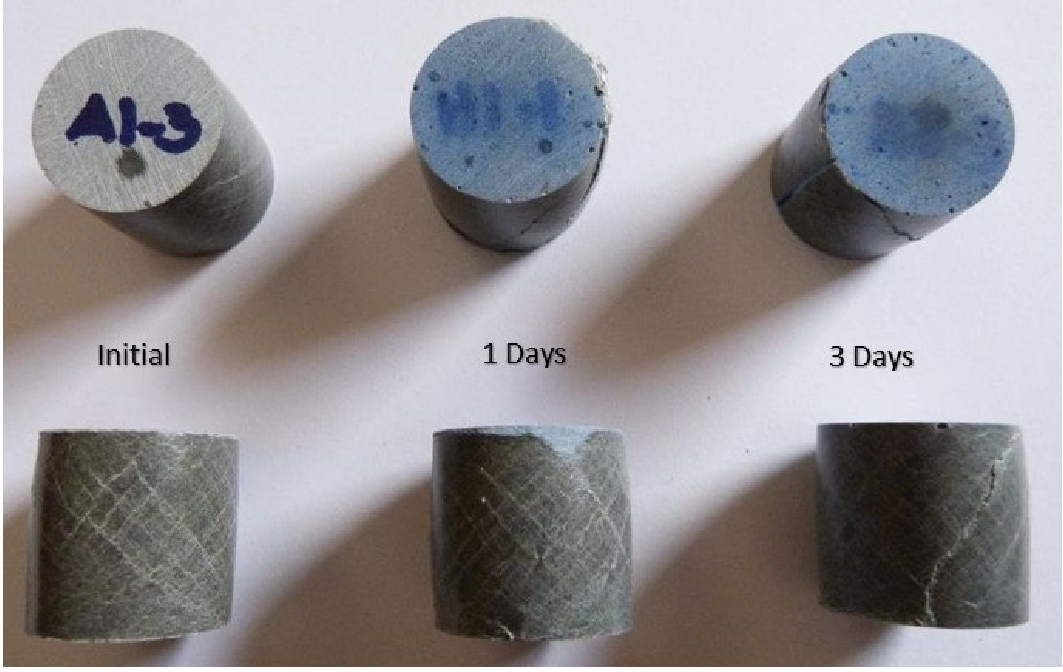

Figure 5.17: Hygrothermal Environment Compression Samples at Increasing Duration (Initial to 3 Days)

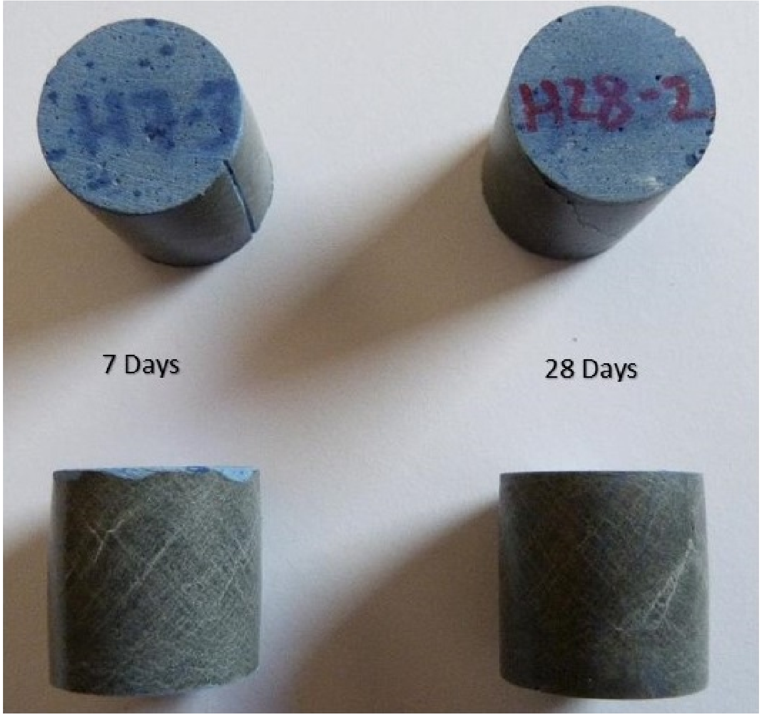

Figure 5.18: Hygrothermal Environment Compression Samples at Increasing Duration (7 to 28 Days)

To emphasise how exposure to the hygrothermal environment affected the compressive failure of the samples a close up comparison between the initial and 28 days is provided in Figure 5.19. After the 28 days the surface of the samples doesnt have any voids, although upon close inspection there are blue lines on the surface. The lines dont follow any pre-existing marks that could have increased the absorption of the blue stained water. Therefore these lines suggest points of water absorption. Apart from the water absorption there are no signs that the shear failure of the samples have changed.

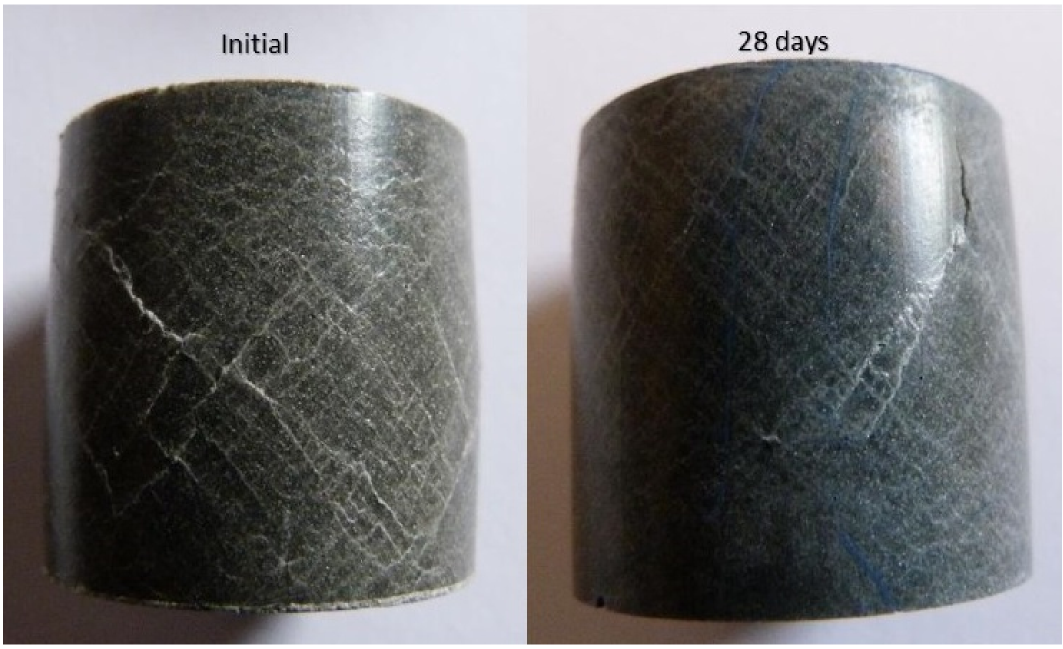

Figure 5.19: Comparison Between Initial and 28 Days Compression Testing for Hygrothermal Environment

MICROSCOPIC OBSERVATIONS

The microscopic observations for specimens exposed to the hygrothermal environment were performed for the whole duration. When comparing the 28 day sample observation (Figure 5.20) to the initial sample (Figure 5.4), though, the same observation was noticed. That observation was that there was no discernible differences. The 1 to 7 days results are provided in Appendix D.

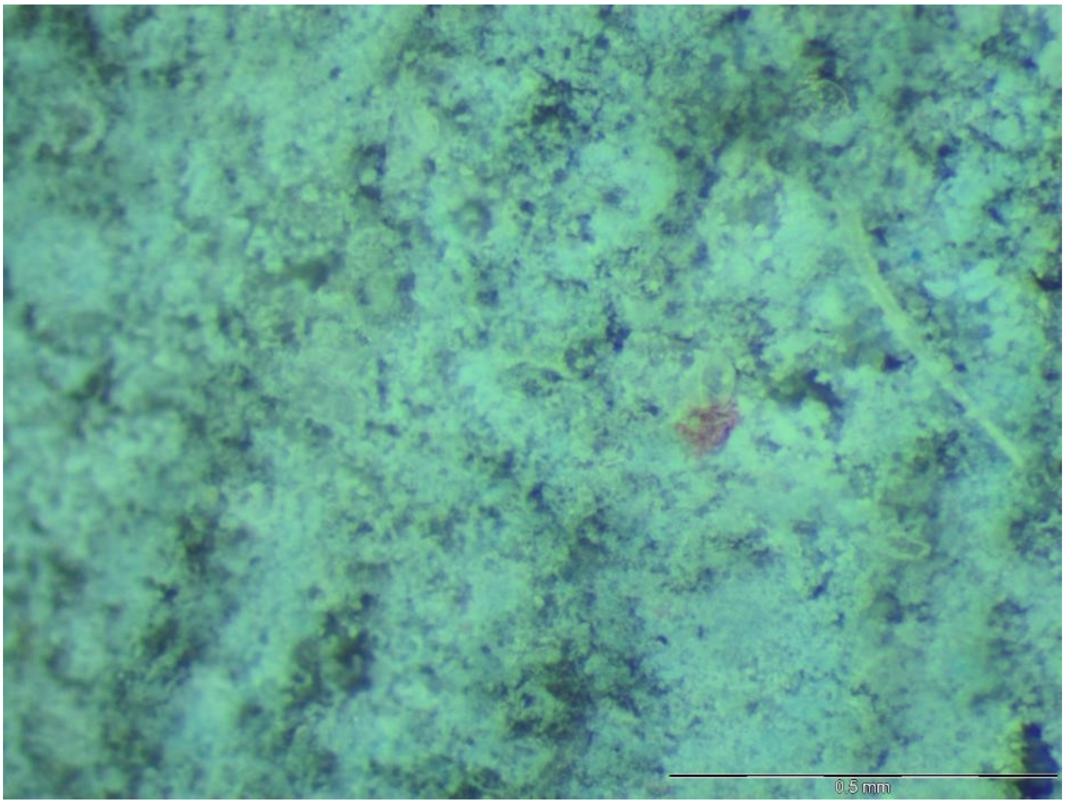

Figure 5.20: Microscopic Observation of 28 Day Sample Exposed to Hygrothermal Environment

Quantitative Results

This section contains the quantitative results for the compression testing, dimension and weight changes (Table 5.4). Figure 5.21 shows the stress-strain behaviour under the compression testing on the specimens exposed to Hygrothermal environment. With the increase in duration the peak stress and modulus of elasticity appear to have increased.

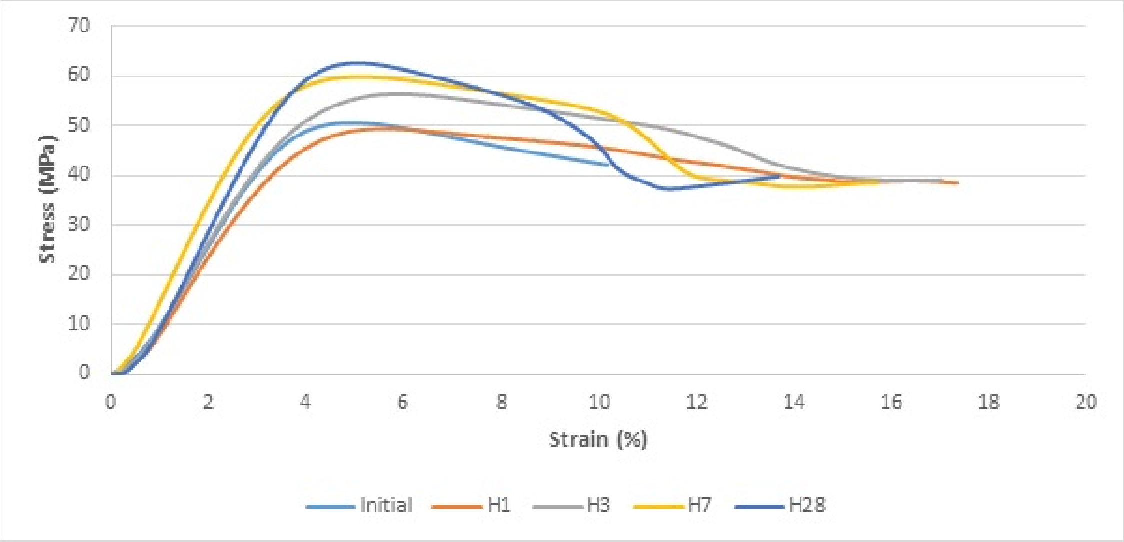

Figure 5.21: Stress vs Strain Graph for Specimens Exposed to Hygrothermal Environment

Due to the inconsistency in exposure to a hygrothermal event, the hygrothermal samples were measured at 2, 4 and 6 hrs after exposure. The specimens exposed to the other environments were not measured at those time periods. From Table 5.4 there is no clear trend for duration and the parameters diameter, height and weight. Although the weight of the specimens was overall increased. With the increase in duration the specimens had an initial decrease in peak load, peak stress and youngs modulus. After the first day, though, there was an increase in all three parameters.

Table 5.4: Quantitative Results for Specimens Exposed to Hygrothermal Environment

Day	Diameter	Height	Weight	Peak Load kN	Peak Stress MPa	Young's Modulus MPa
0	1	1	1	34.53	50.03	1573.7
0.083	1.00170	0.99222	1.00063	-	-	-
0.167	0.99679	0.99026	1.00052	-	-	-
0.25	0.99108	0.98815	1.00048	-	-	-
1	1.00271	1.00227	1.00060	34.36	49.86	1463.7
3	1.00175	0.99967	1.00170	38.89	56.58	1643.9
7	1.00011	0.99330	1.00210	40.96	59.89	1858.4
28	1.00169	0.98879	1.00184	42.57	61.81	1926.0

5.3 Discussion

5.3.1 Effects of Duration

This sections analyses the effects of duration for each individual environment.

Air Environment

With the increase of duration a couples of trends were noticed for the specimens exposed to air. The first was that the peak load, peak stress and youngs modulus all increased with duration. This suggests that the specimens were becoming stronger with time. From Figure 5.3 would confirm this as there was less distribution of the shear cracks. According to Lokuge & Aravinthan (2013) the compressive strength gain is negligible, which doesnt support the results. Instead the results support Custdio et al. (2011), which stated that curing at ambient temperature results in only partially cured samples. Therefore the specimens would be continually curing with the duration. This brings into question how long the samples take to cure when the increase in compressive strength continued to the six month mark.

Secondly, in Table 5.1 it was pointed out that there was no trend for the parameters diameter, height and weight. With the constant fluctuating of the values for the diameter and height, the changes calculated would most likely be the result of human error in the measuring. When the height and diameter were being measured, it was aimed that these parameters were measured from the same spot to get an accurate measured change. It is possible, though, that the measuring could have been performed in a slightly different area resulting in the fluctuating.

For the weight measurements, the scales used provided more accuracy, as can be noted by the consistent results. On the 121 day, though, there was a spike of a 1% increase in weight. This was followed by a decrease to the original values of 0.003%. The 121 value is being considered an anomaly caused by human error. Reasons for the anomaly include recording the wrong number or not tarring the scales properly before measuring.

With the errors in mind the parameters, diameter, height and weight are considered to have stayed constant throughout the duration of being exposed to air. This was to be expected, as the control, as epoxy has minimal cure shrinkage (Lokuge & Aravinthan 2013). The inclusion of the hollow microspheres also reduces the shrinkage of the specimens (Aruniit, Kers, Majak, Krumme & Tall 2012).

Water Environment

With the increase of duration a couples of trends were noticed for the specimens exposed to water. The first was that the weight, peak load and peak stress increased with duration (Table 5.2). At the 168 day mark, though, there was no increase for the peak stress and a slight decrease for the peak load and weight. The slight decrease in weight could have been the result of degradation of the samples resulting in voids or over drying the samples.

Despite the decrease in weight gain, the samples did absorb water with a 0.3% weight gain. When compared to the experiment performed by Alamri & Low (2012), the epoxy resin had a weight gain above 2%. In that experiment they used FR-251 epoxy and the samples were submerged in room temperature water for 130 days. Alamri & Low (2012) concluded that filler significantly reduced the water absorption of the specimens, which explains why weight gain was significantly lower than 2%. The weight gain was also compared to the results from Hu, Shan, Zhao & Tong (2015), where using post cured DGEBA, there was a weight gain of 0.8% after 5 days. This value is closer to the 0.3% from Table 5.2, which is to be expected as the only difference was that the samples were post cured. Hu et al. (2015) also concluded that the addition of fibres reduced the water absorption. Therefore the addition of filler reduced water absorption of the samples.

Despite the addition of filler decreasing water absorption; flexural strength and modulus decreased with water absorption (Alamri & Low 2012). At the same time the addition of filler increased the fracture toughness and impact strength. Although too much of certain fillers, like nanoclay, proved the opposite for fracture toughness and impact strength. This statement explains the results in Table 5.2, as the compressive values increase with water absorption. The plateau of that increase at 168 days, though, cannot be explained as the information about the filler was only tested to 130 days (Alamri & Low 2012). This suggests that there is a limit to the increase in compressive values proportional to the water absorption, as the values dropped with the water absorption. Another possible explanation is that the water started to degrade the surface of the samples, which would have resulted in plans of weakness.

When comparing the results to Figure 5.9, after the 168 days there was an increase in large voids on the surface of the sample. This could have resulted in an increase water absorption, although being on the surface the water would have been removed upon drying, resulting in the drop in weight. The voids would have also contributed to the slight drop in the peak load and plateau of peak stress, as it added a plan of weakness.

Secondly, in Table 5.2 it was pointed out that there was no trend for the parameters diameter and height. This trend, as previously mentioned in the previous section, would have been the result of human error in the measuring. For the youngs modulus there was also a fluctuation with the value with increase duration. It reached its peak value at 28 days, which was much shorter that the other values derived from the compression testing at 121 days. A possible explanation is the one used above, that the samples were only partially cured and continued to gain compressive strength with time. After the 28 days though there was the decrease in value. This drop can be explained by Alamri & Low (2012), who found that water absorption decreased flexural modulus by approximately 200 MPa which was similar to the drop noticed in Table 5.2. When the weight dropped, and thus water absorption there was a slight increase in the youngs modulus.

Salt-Water Environment

With the increase of duration a couples of trends were noticed for the specimens exposed to salt-water. The first was that the weight, peak load and peak stress increased with duration (Table 5.3). At the 168 day mark, just like for the specimens exposed to the water environment, the value decreased for peak load and peak stress. The weight of the samples, however, continued to increase.

Similarly to the water samples, the samples exposed to the salt-water environment had a 0.3% weight gain. With the only difference between the water and salt-water environments being the salt it is expected that the results between the two environments would be similar. Due to the samples not decreasing in weight at the 168 day mark, it suggests that large voids were not present on the surface of the sample, as the samples retained the weight. Despite the weight increasing though, the values of peak stress and peak load still declined. While, the decline in values is considered minor, it does suggest that the samples were weakened.

When comparing the results to Figure 5.14, there was an increase in voids. These voids were small in size allowing the sample to still retain the water after drying the surface. The voids would have still created a plan of weakness resulting in the drop in values.

Secondly, in Table 5.3 it was pointed out that there was no trend for the parameters diameter and height. This trend, as previously mentioned in the analysis of the air and water environment, would have been the result of human error in the measuring.

For the youngs modulus there was also a fluctuation with the value with increase duration. At first there was a 200 MPa decrease after 7 days, which was unexpected. After this the value increased above the initial value at the 28 day mark. Youngs modulus represent the stiffness of the specimen, and isnt the most accurate measure of properties of the specimen. At the 7 day mark there was an increase in peak load and peak stress, suggesting an overall increase in strength, while the decrease in modulus suggest that the specimen has become less stiff. Due to the inaccuracy of using the youngs modulus, the decrease in value is considered an anomaly due to the 500 MPa increase at the 28 day mark.

The youngs modulus reached at peak at the 56 day mark, after which there was a steady decrease. Again this can be explained by Alamri & Low (2012), who found that there was a decrease in flexural modulus with increase in water absorption. This decrease in value also increased with the increase in water absorption.

Hygrothermal Environment

With the increase of duration a couples of trends were noticed for the specimens exposed to the hygrothermal environment. The first was that the weight, peak load, peak stress and youngs modulus increased with duration (Table 5.4). When compared to the initial results, for the 7 day result there was a decrease in values for the peak load, peak stress and youngs modulus. This initial decrease was also noted for the youngs modulus of the salt-water samples. Although, with minimal decrease for all values, it most likely resulted from minor differences in the samples not the exposure. The weight of the samples did fluctuate, with there being a decrease at the 169 day mark, however, there was always water absorbed.

Despite the decrease in weight gain, the samples did absorb water with a 0.2% weight gain. When compared to the experiment performed by Srihari, Revathi & Rao (2002*a*), the epoxy resin had a weight gain of 1.64%. For this experiment they used LY5052 epoxy resin with glass composites, post cured the samples and the sample were a 25mm x 25mm x 2mm size. The experiment tested the effect of immersion in water and salt-water, both at 60°C. Comparing the tile shape of the sample to the cylindrical shape used in Study 2, the tile shape has a significantly larger surface area to volume ratio. This allows the for greater moisture absorption, which could explain why their result was almost 10 times the results in Table 5.4. The study also compared the result to neat epoxy, which absorbed around 4.5%. From this result it can be concluded that the filler has reduced the water absorption of the specimens.

In a separate experiment Srihari, Revathi & Rao (2002*b*) determined that with water absorption there was a decrease in compressive strength and hence stiffness. This statement doesnt match the results as there was an increase in values with duration. A possible explanation is that while being exposed to water above 60°C, the samples were post cured. Post curing involves exposing the specimen to elevated temperatures for a set time and increases mechanical properties and glass transition temperature. From Study 1, the epoxy resin used has a glass transition temperature between 50-60°C. Therefore the hygrothermal environment is exposing the specimens to an elevated temperature to induce post curing. Srihari et al. (2002*b*) had already post cured there sample, so they were expected to have the maximum mechanical properties they could get. Therefore, the result of water absorption reduced those properties.

When comparing the result to Figure 5.19, after the 28 days there was no presence of voids on the surface of the specimen. There was, however, clear sign of water absorption with the blue staining of the sample. Due to there being no voids, when compared with the water and salt-water results, it either suggest that post curing had occurred reducing degradation or that exposure was too short for degradation.

Secondly, in Table 5.4 it was pointed out that there was no trend for the parameters diameter and height. This trend, as previously mentioned in the rest of Section 5.3.1, would have been the result of human error in the measuring.

5.3.2 Effects of Environment

This section analyses the effect of environment on each individual parameter.

Physical Observations

Figure 5.22 to 5.26 illustrates the comparison between the compression tested samples of all environments at each testing period.

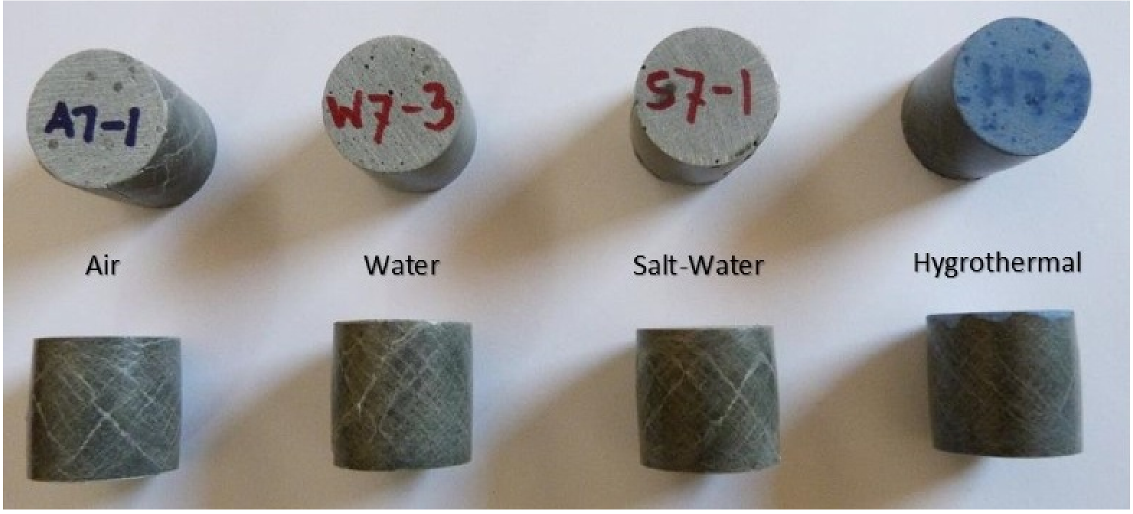

Figure 5.22: Compression Samples at 7 Days

After the 7 day testing, the compressive failure of the specimens is similar for each environment (Figure 5.22). The shear failure is uniform across the surface of the specimens. The only difference is the blue staining of the hygrothermal specimens. This, however, was the result of using a non-permanent marker when labelling.

Figure 5.23: Compression Samples at 28 Days

After the 28 day (Figure 5.23) testing, the specimens showed the same result as the 7 day results, with uniform shear cracking. The 28th day was the last day that the hygrothermal environment was tested. Based on physical results along, the hygrothermal environment did not affect the specimens in a different way compared to the other environments.

Figure 5.24: Compression Samples at 56 Days

After the 56 day (Figure 5.24) testing, the compressive failure has resulted in more pronounced shear failure along one plane. In the water sample the shear failure was not as pronounced, this would have been the result of the large voids that have become present on its surface. These voids provide planes of weakness, which results in pronounced failure branching out from the voids. The specimens exposed to the salt-water dont show signs of voids in the surface, which is why there is a more pronounced shear failure similar to the specimen exposed to air.

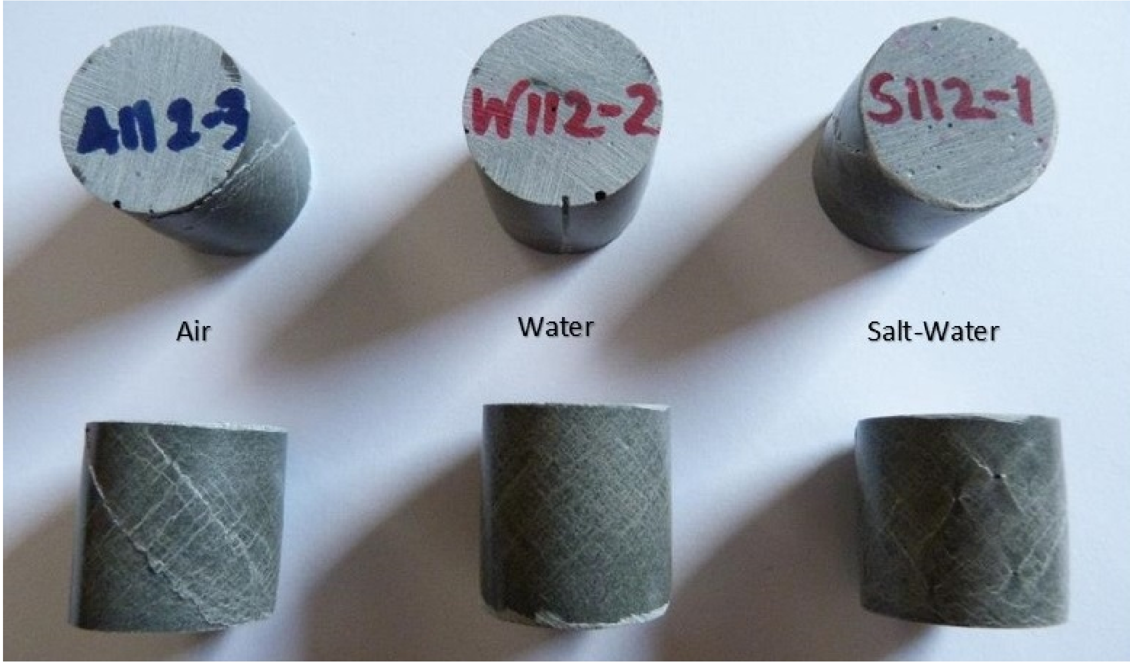

Figure 5.25: Compression Samples at 112 Days

After the 112 day (Figure 5.25) testing the specimen exposed to air is showing more pronounced signs of shear failure. For the specimens exposed to the water and salt-water environments there are little signs of pronounced failure. Instead for the water environment the failure is uniform across the surface, as there are no large voids present. Large voids are present on the surface and top of the specimen exposed to the salt water. This resulted in more distributed shear failure that is pronounced around the voids.

Figure 5.26: Compression Samples at 168 Days

The 168 day (Figure 5.26) was the last day of testing for the rest of the samples. The specimens exposed to the air environment had a pronounced shear failure, with very little distribution. The specimens exposed to the water and salt-water environments also showed this trend. From Figures 5.9 and 5.14, though, a different side of the sample was shown. In Figure 5.9 there were large voids present in the water specimen and the pronounced failure did not follow one plan of weakness. Instead towards the bottom and top of the sample the shear failure travelled more vertical. The shear failure was also more distributed when compared to the specimen exposed to air. In Figure 5.14 only small voids were present in the salt-water specimen. Even though these voids were smaller they still resulted in a more distributed shear failure similar to the water specimen.

From the physical observations the specimens exposed to air have increased pronounced shear failure with increase in duration. For the specimens exposed to either water or salt-water the environments, due to degradation, more voids are present on the surface with duration. Irrespective of size, the voids provided plans of weakness that result in more distributed shear failure. The specimens exposed to the hygrothermal environment showed no properties that were different from the other specimens at the end of its testing. This is either the result of the environment providing no changes to the samples, or that a longer duration was needed to see sign of degradation.

Load Deformation

The three values that were determined from the compression testing were peak load, peak stress and Youngs modulus. Figure 5.27 to 5.29 illustrate the trend analysis of those parameters respectively.

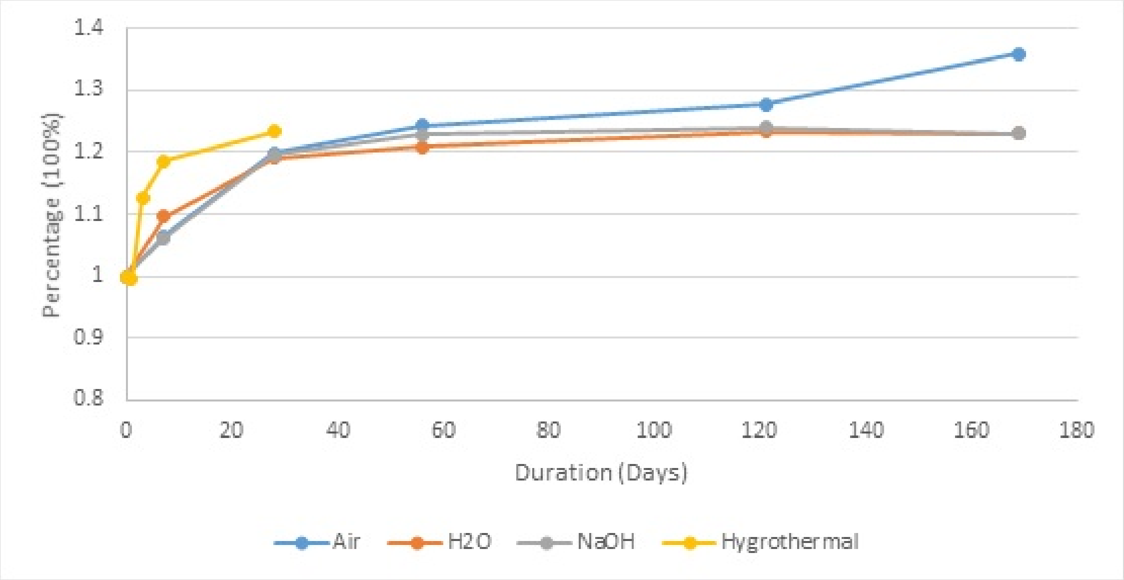

Figure 5.27: Trend Analysis of Peak Load Over Duration

For the peak load the specimens exposed to the hygrothermal environment had a more immediate increase in value. At the 7 day mark it had an approximate increase of 20%, while all three of the other environments had an increase of around 10%. The biggest difference between the hygrothermal environment and the other three was the exposure to the elevated temperature. Even at the 28 day mark, which was the last testing day for the hygrothermal environment it still had the highest increase. Due to the temperature being the key difference that resulted in the greatest increase, it suggests that the specimens were post cured in the hygrothermal environment. For the comparison between the other three environments they all had a steady increase to the 121 day mark. After this the air continued to increase, while the water and salt-water specimens had a slight decrease in values. This first suggests that the samples were only partially cured as the values continued to increase for the air environment. Secondly that the salt does not affect the how the water reacts with the samples.

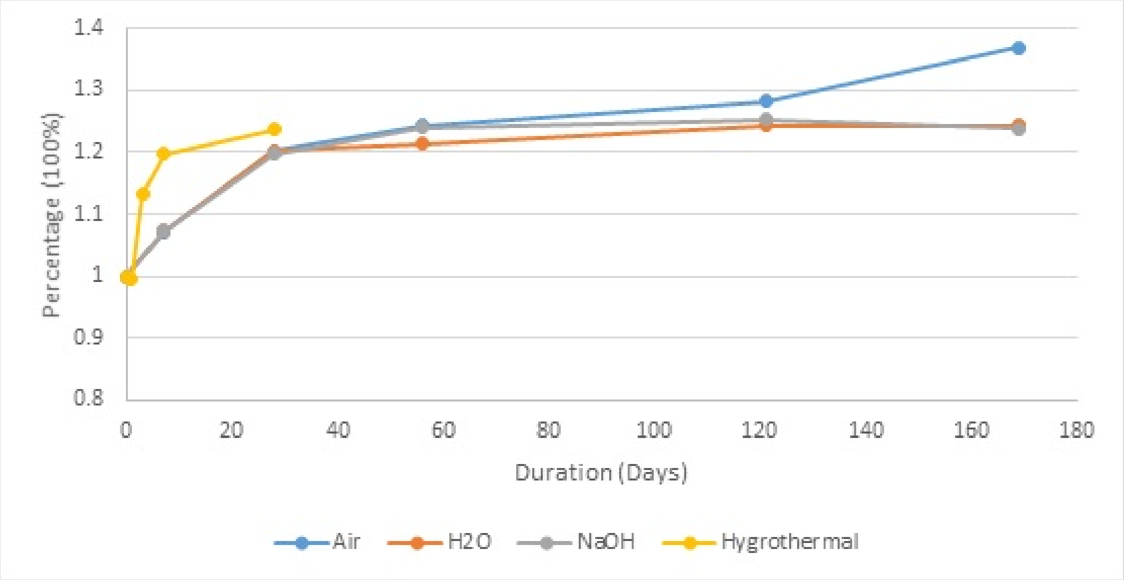

Figure 5.28: Trend Analysis of Peak Stress Over Duration

For the peak stress the exact same trend noticed for the peak load is seen. Firstly that the hygrothermal environment had a more pronounced increase in values, suggesting that the samples was post cured. Secondly that the sample may have only been partially cured as the samples exposed to air had a steady increase in values. Finally that the water and salt-water specimens had the same trend of an increase to the 121 day mark followed by a slight decrease. This suggested that the salt had no detrimental effect on the epoxy sample, as epoxy is chemical resistant.

Reis (2009), however, determined that salt-water resulted in 4% more compressive strength loss when compared to distilled water, which was 0.7%. In their experiment though the sample were exposed to degradation cycles, not immersion. The sample were also post cured instead of cured at ambient temperatures. The biggest difference was that in Study 2 tap water was used instead of distilled water. Tap water has more impurities, which simulates the possible water environments the epoxy may be exposed to. Distilled water is pure water, which the epoxy is unlikely to be exposed to. Despite the 4% difference in compressive values, when looking at the results for Study 2 the epoxy samples have maintained chemical resistance.

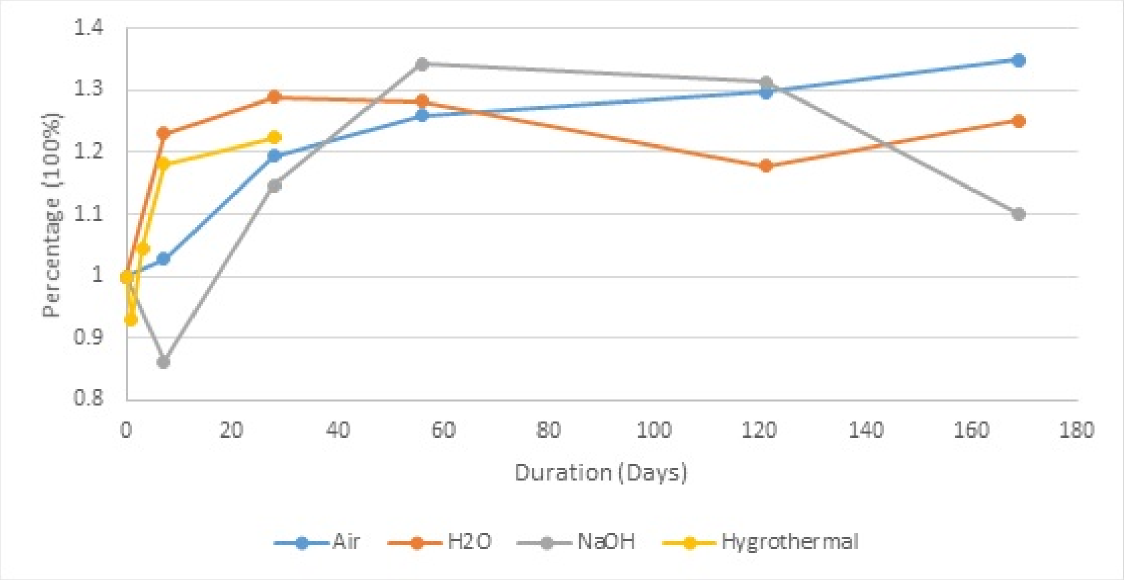

Figure 5.29: Trend Analysis of Young's Modulus Over Duration

For the Youngs modulus the values fluctuated, with the specimens exposed to air being the only environment to show a consistent increasing trend. This would be expected as with the increase in strength, stiffness is often also increased. For the other environments though, there were decreases in stiffness that were not related to a decrease in compressive strength. This could have been the result of impurities in the samples or the number of voids present in later specimens. Alamri & Low (2012) suggested that a decrease in the modulus was related to the increase in water absorption. The initial increase of youngs modulus could be contributed to the samples not being post cured, allowing increase in strength before the environments started to degrade the samples. This aligned with the fluctuation of the weight, except for the initial drops in values for the hygrothermal and salt-water environment. Due to the inaccuracy of using youngs modulus and the consistent trend for the rest of the value, this initial decreases are considered anomalies.

Overall though despite water absorption, at the end of the testing period all sample had an increase in youngs modulus when compared to the initial value. This may have decreased below that value with longer exposure.

When looking at the values for Youngs modulus in Table 5.1 to 5.4 it is noted that the values are on average 400 MPa higher than 1371 MPa, which was the highest value recorded for Study 1. It was mentioned in Problems in Section 4.3.3 that the lumps would have resulted in changes to the youngs modulus. The average increase in values for Study 2, which had the lumps removed before the mixing process, proves that the lumps affected the youngs modulus.

Dimensions

In Section 5.3.1 it was pointed out that there was no trend for the dimensions, diameter and height. There was constant fluctuating of values in all environments. This can be seen in Figure 5.30 and Figure 5.31 which illustrate the trend analysis of the diameter and height changes respectively.

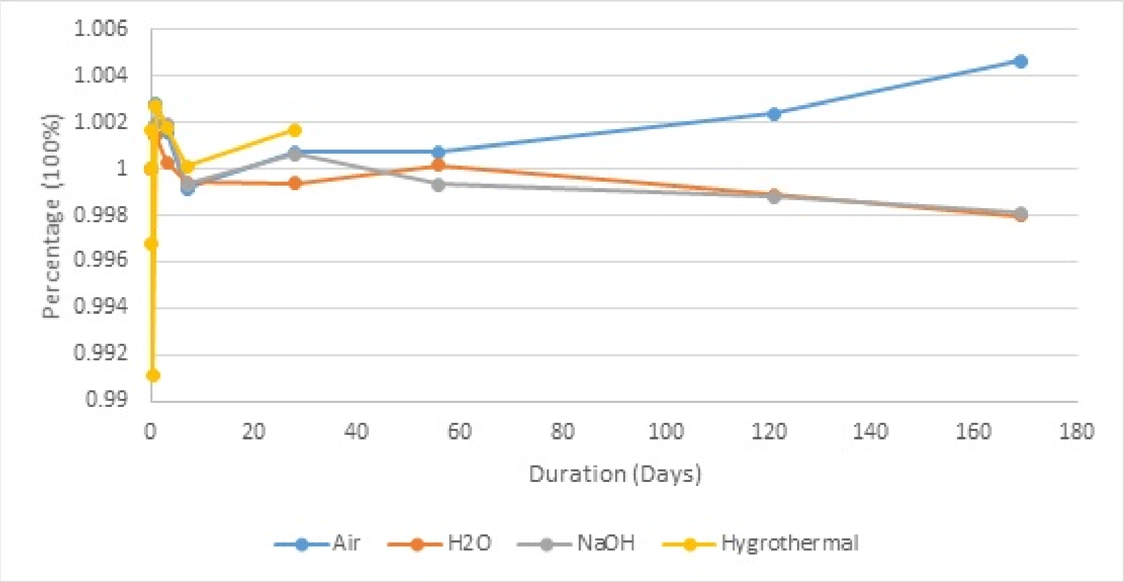

Figure 5.30: Trend Analysis of the Diameter Over Duration

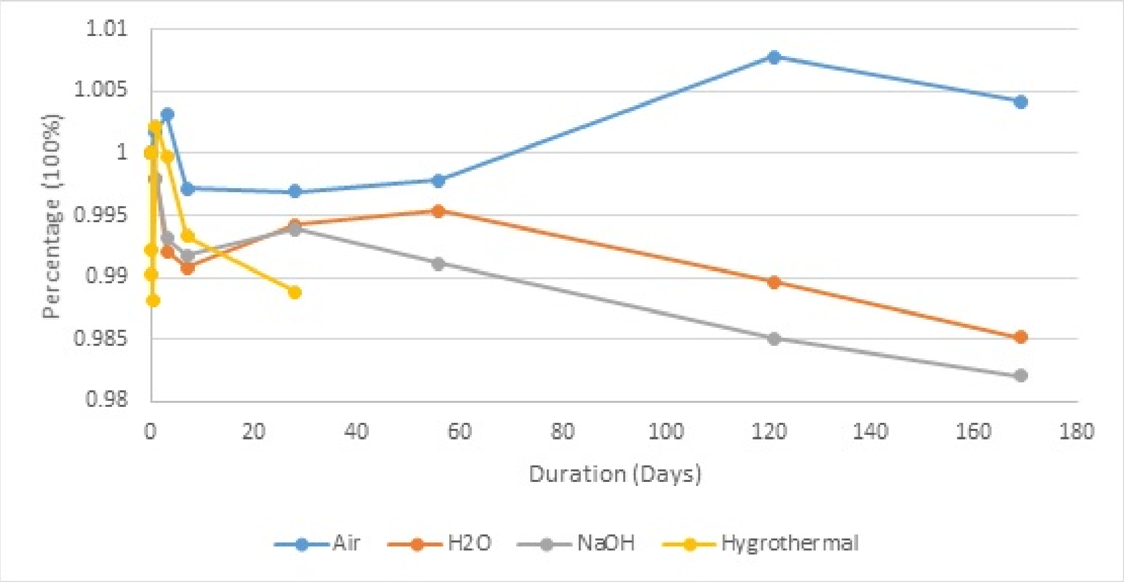

Figure 5.31: Trend Analysis of the Height Over Duration

Constant fluctuating between environments would be the result of human error in the measuring. This is either by recording the wrong number or by measuring in a different manner to how it was done previously. Due to the constant fluctuating, the results are considered as there was no change in the dimension. No change in the dimension, mean that the specimens did not shrink due to curing or swell due to water absorption. This is to be expected, as mentioned in Section 5.3.1 the epoxy resin has minimal cure shrinkage with the hollow microsphere provided more shrink resistance.

Weight

Figure 5.32 illustrates the trend analysis of the weight changes with the increase in duration. For the samples exposed to the hygrothermal, water and salt-water environments, there is a steady increase in weight with the increase in duration. For the samples exposed to air, due to the 121 day test being excluded as mentioned in Section 5.3.1, the weight remains constant. Looking at Figure 5.32 it illustrates how much of an anomaly the 121 day air value was.

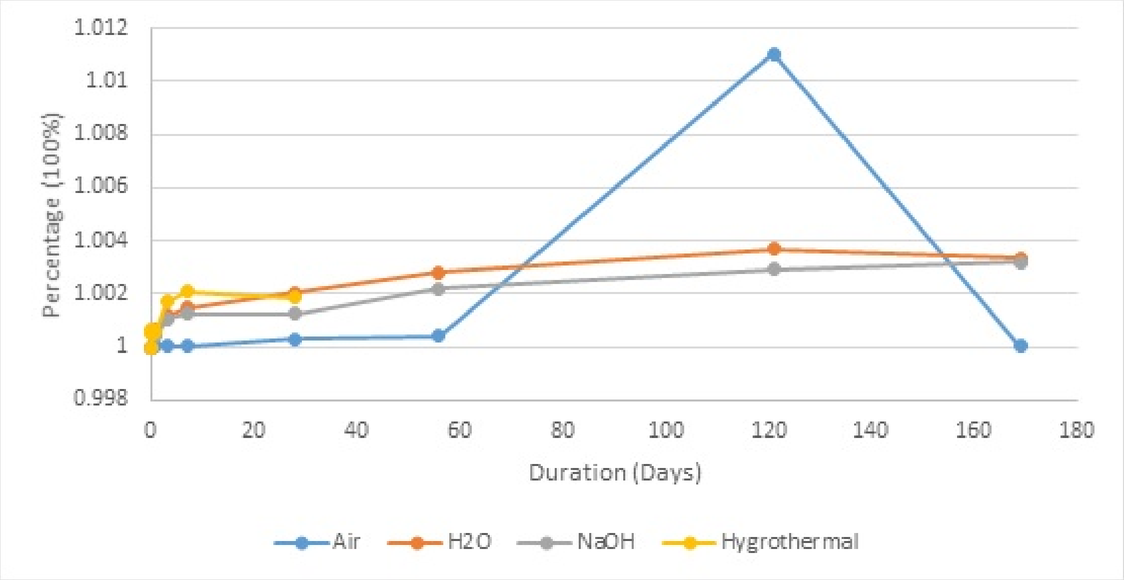

Figure 5.32: Trend Analysis of the Weight Over Duration

From the above section it was determined that the water absorption was causing any swelling in the samples. This would mean that the water absorption would be the result of voids present in the surface of the specimens. The advantage of this is that the water absorption is limited by the number of voids present in the surface. However, in the Physical Observations of Section 5.3.2 it was noted that long exposure to water and salt-water environments resulted in degradation with additional and larger voids. If the duration continued it could be determined if the degradation and thus water absorption continues or reaches a limit.

Epoxy resin is known for low water absorption (Lokuge & Aravinthan 2013), despite the addition of filler that fact remain true. This is because the samples did not absorb more than 0.4% after 169 days. This aligns with the information provided by Kirlikovali (1981) in Chapter 2, that polymer concrete has a water absorption of 0.3%. Therefore exposure to different environments doesnt increase the water absorption of the epoxy samples after 169 days.

Microscopic Observations

In Section 5.2 it was mentioned for each environment that the microscopic observation did not show any changes with duration. Therefore there are no results to compare and it is suggested that a stronger magnification is required.

5.3.3 Limitations and Problems

Limitations

In Study 2 a major limitation was found with reference to the use of the microscopic observations, which was previously mentioned in the Microscopic Observations above. No discernible differences were present between the initial specimen and the last specimen observed. Due to no results the microscopic observations stopped after the 112 day samples. It is suggested that a stronger magnification is required to determine changes to the epoxy matrix. This is proven by comparing Figure 5.4 with a scale of 0.5mm (500 μm), to Figure 5.33 with a scale of 200 and 20 μm (Park et al. 2005). With the increased magnification of the sample in Figure 5.33 the filler particles can be clearly seen. From this magnification, a more concrete analysis can be provided to determine the effects the environment had on the epoxy matrix.

The limitation that was faced was Figure 5.4 was the strongest magnification possible with the optical microscope available at the University of Southern Queensland. Therefore a stronger optical microscope would be required to be sourced and then negotiations would be required for the use of it. This was impractical due to the limited time frame and the results obtained from the other parameter were sufficient for a summary of the effect of the environment. Also the microscopic image had to be taken of a flat surface and the only flat surfaces on the sample were the two ends. The problem with the ends was that the ends had been sanded flat, which could cause irregularities between the results. Therefore it was decided that the microscopic observations wouldnt be used to make a conclusion on the effect of environment on the epoxy specimens.

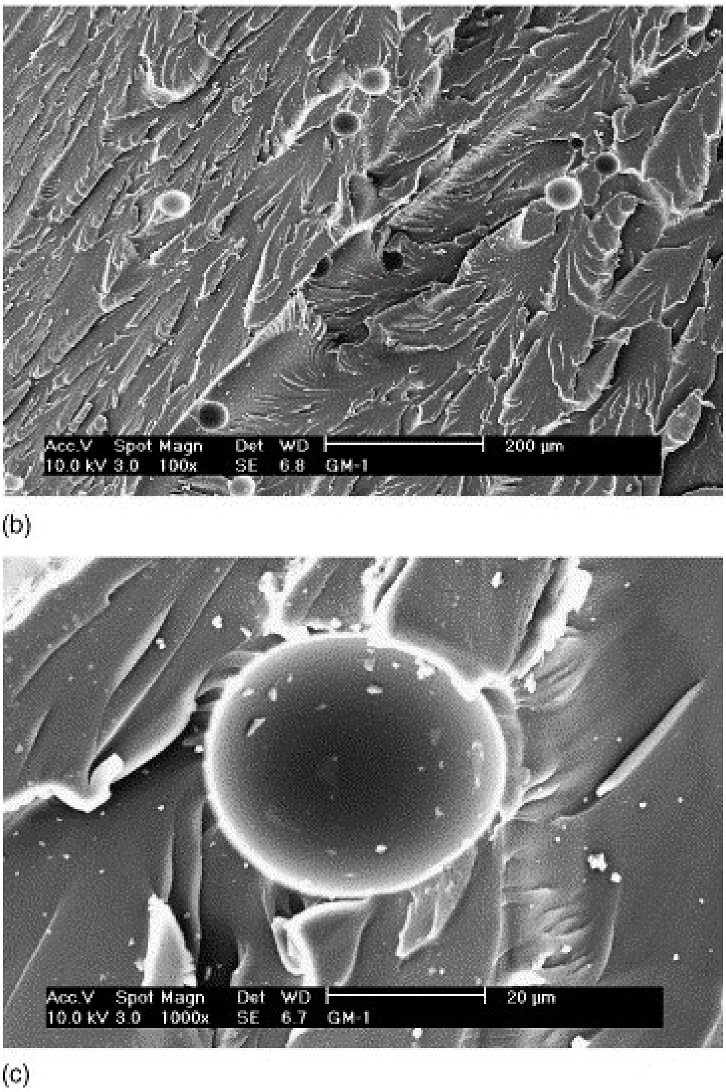

Figure 5.33: SEM Micrographs of Epoxy Composites After Fracture Toughness Test (B) 1 wt. % Filler (Magnification of 100) and (C) 1 wt. % Filler (Magnification of 1000) (Park et al. 2005)

Problems

In Study 2, two problems were encountered that would have altered the experimental results. The first was mentioned in Section 5.2.1, about how the 112 and 168 day tests were actually performed on the 121 and 169 day. To accurately represent the trend analysis with increasing duration the actual days of testing were used in the graphs. In the figures, however, the samples had already been labelled by the day they were supposed to be tested on. This could lead to confusion with the changing numbers for reference. Apart from that it wouldnt affect the results in any other way.

The second was that the hygrothermal samples were removed from their environment before the full 28 day period. On the 28 day testing I was informed that the hygrothermal samples had been removed from their environment earlier that week; exact date unknown. The samples had been labelled with a contact number provided, although, someone else required the stove and removed the samples. Due to the date of their removal not being known, it is unknown how this may affected the results for the hygrothermal environment.

For the 28 day test results there was an increase in the values for peak load, peak stress and youngs modulus which followed the trend set by the preceding results. There was, however, a decrease in water absorption which didnt follow the trend. From the preceding results it had been determined that exposure to the elevate temperature was post curing the sample and increasing the mechanical properties. The 28 day test result could have determine whether the values increased at the same rate, slow down as it reaches a limit, or reacted with the water and start to degrade the specimens. If degradation occurred it could have also been compared to the results of the water and salt-water environments, to determine if it results in voids in the surface or if overall degradation is accelerated due to the temperature.

Despite the 28 day test results not being accurate, based off the initial 7 day results it was determined that the temperature was increase the mechanical properties at an accelerated results, by providing a means for post curing.

5.4 Summary

Chapter 5 evaluated the six month durability of the optimal mix design, determined in Chapter 4, with respect to simulated environmental conditions. The environmental conditions were air, water, salt-water and hygrothermal. The specimens exposed to air maintained consistent dimension and weight. With the increase of duration the peak load, peak stress and Youngs Modulus also increased, resulting in more pronounced compressive shear failure.

The specimens exposed to the water and salt-water environments presented the same trend with the increase in duration. Approximately 0.3% of water was absorbed for specimens exposed to both environments. This aligned with the water absorption for neat polymer concrete, proving that the filler had no effect on the water absorption of the epoxy. The dimensions stayed the same despite water absorption, which were absorbed by voids present in the surface of the samples. With water absorption compressive values increased, however, the water also deteriorated the surface resulting in more and larger voids in the surface. These voids weakened the sample, resulting in decrease in the compressive values at the six month testing.

For the specimens exposed to the hygrothermal environment, the elevated temperature resulted in post curing. Post curing accelerated the increase in mechanical properties. After the 28 day testing, which was the limit for the hygrothermal environment, there was no decrease in properties or signs of deterioration on the surface on the sample. The dimensions stayed the same and there was approximately 0.2% water absorption.

From Chapter 5, it can be concluded that the optimal mix design meets six month durability exposed to simulated environmental conditions. The filler had no adverse effects on the durability of the specimens. Deterioration resulting in voids was the major cause of weaknesses in the samples.

Chapter 6

Conclusion and Recommendations

6.1 Conclusion of Results

The aim of this project was to analyse the effect light-weight particulate filler has on the thermomechanical and durability properties of epoxy resin. To achieve this aim the project consisted of two experiments that focused on the effect of the filler on each property of the resin.

The first experiment consisted of epoxy based polymer matrix with different percentages of filler. These specimens was then tested under compression in an environmental chamber at room temperature, 30, 40, 60 and 80°C.

The second experiment consisted of exposing the optimal epoxy resin to filler mixture to simulated environments. These specimens were exposed to either air, water, salt-water up to 6 months or hygrothermal environments up to 1 month.

The aim and results of each experiment is presented below.

6.1.1 Behaviour of Epoxy-Based Polymer Under Elevated Temperature

Chapter 4 determined the optimal filler content, with respect to elevated temperature, that had the least reduction in compressive strength of the epoxy resin. Based on the results of this study, the following conclusions can be drawn:

- The epoxy-based polymer resin with 60% filler by weight were least effected by the increase in temperature. There was an overall decrease of 85.2% and 92.1% in peak stress and Youngs modulus respectively. However, this mixture can create issues in actual application due its low workability, voids and brittle compressive failure.

- The epoxy-based polymer resin with 20% filler by weight had an overall decrease of 92.8% and 98% in peak stress and Youngs modulus. Due to the low density of the mixture, lumps settled to one end. Compressive failure resulted in fracturing in the end that contained the lumps.

- The epoxy-based polymer resin with 0% filler by weight had an overall decrease of 91.5% and 98.7% in peak stress and Youngs modulus. Similar to the specimens with 20% filler, lumps settled to one end. Compressive failure was bulging with fracturing in the end that contained the lumps.

- The epoxy-based resin with 40% filler by weight were the second least effected from the increase in temperature. There was an overall decrease of 90.2% and 96.4% in peak stress and Youngs modulus respectively. Minimal voids were present on the specimens and compressive failure was an evenly distributed shear failure. Based on this it was concluded that the optimal mix design was determined to be the 60:40.

6.1.2 Six-Months Durability Test

Chapter 5 evaluated the six month durability of the optimal mix design, determined in Chapter 4, with respect to simulated environmental conditions. The environmental conditions were air, water, salt-water and hygrothermal. Based on the results of these studies, the following conclusions were drawn:

- For the specimens exposed to the air environment, dimensions and weight remained consistent. With the increase of duration there was an increase of 38% and 35% in peak stress and Youngs modulus respectively.

- For the specimens exposed to the water and salt-water environments the same trends were presented with the increase of duration. Therefore the salt had no effect on the durability of the specimens. Dimensions remained consistent despite 0.3% water absorption. With the increase of duration there was an increase of 24% in peak stress and no trend was present for Youngs modulus.

- For the specimens exposed to the hygrothermal environment, the elevated temperature resulted in post curing. After 7 days exposure, the peak stress had an increase of 20%, which was 12% higher than the specimens exposed to the other environments. Dimension remained consistent with 0.2% water absorption. Like the specimens exposed to the water and salt-water environments, no trend was present for Youngs modulus.

From Chapter 5, it can be concluded that the optimal mix design meets six month durability exposed to simulated environmental conditions. The filler had no adverse effects on the durability of the specimens. Deterioration resulting in voids was the major cause of weaknesses in the samples.

6.2 Recommendations

In this section recommendations are formed from the findings. These recommendations include those to improve the design of the study, recommendations for the application of the results and future works.

6.2.1 Improvements

From Chapter 4 the only problem that would require rectifying would be the Part A lumps within the mixture. The improvement to the experiment to rectify this problem would be to sieve the lumps out of the material before weighing the amount required. This ensures that the lumps are not present within the mixture.

From Chapter 5 there are two recommended improvement. Firstly to ensure that the specimens are not removed prematurely from the simulated environments. This could be done by providing easily accessible information about who to contact and also the length of time that the equipment will be used. The second improvement would be to gain access to a more powerful microscopic. This would allow microscopic results that could be analysed.

6.2.2 Recommendations

Based on the results of the two experiments the following recommendations have been made.

- Ensure all material are uncontaminated and of even consistency before use.
- The service temperature has to be determined before application to ensure that the right epoxy is used.
- Mixtures with less than 50% epoxy results in voids and brittle failure, therefore, it is not recommended to be used.
- The optimal mix of epoxy resin and light-weight filler (fly ash, fire retardant and hollow microsphere) is 60:40.
- To further decrease the effects of temperature it should be evaluated if post-curing is economical and feasible.
- Exposure to air, water, salt-water and hygrothermal environments does not negatively affect the durability of the specimens.

6.2.3 Future Work

Apart from redoing the experiments to implement the improvements mentioned in Section 6.2.1, to ensure the validity of the results, possible further works are listed below. These future works aim to further the research of the epoxy resin discussed in this report or are based on gaps in the literature.

- Research and study the effects of temperature on epoxy resin with different filler materials.
- Research and study the effects of temperature on the specimens with varying amounts of fine and coarse aggregate.
- Evaluate several guidelines for the assessment of the glass transition temperature and allowed service temperature and suggest a unified guideline for commercial use.
- Evaluate the effect of a longer exposure period on the optimal mix.

- Conduct field studies to determine actual service temperature and if a hygrothermal environment results in post-curing.
- Evaluate the effect of a dynamic exposure to environments.

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Appendix A

Project Specification

University of Southern Queensland

FACULTY OF HEALTH, ENGINEERING AND SCIENCES

ENG4111/4112 Research Project
PROJECT SPECIFICATION

FOR: **ASHLII TIMMS**

TOPIC: EFFECTS OF ELEVATED TEMPERATURE AND SIMULATED ENVIRONMENTAL CONDITIONS ON THE PROPERTIES OF EPOXY BASED POLYMER RESIN

SUPERVISOR: ALLAN MANALO

ENROLMENT: ENG4111 – S1, ON-CAMPUS, 2015;
ENG4112 – S2, ON-CAMPUS, 2015

PROJECT AIM: To determine the effects of elevated temperature on the compressive properties of epoxy based polymer concrete with different percentages of epoxy resin binder and light-weight particulate filler. Furthermore, an investigation into the effect of exposure to water and saltwater on the durability of the optimised Particulate Filled Resin (PFR) will be conducted.

PROGRAMME: Issue B, 16th March 2015

1. Research the background information relating to epoxy based polymer concrete, light-weight particulate filler.
2. Research on the effects of temperature on the compressive properties of epoxy based polymer resin with different resin to filler content ratio.
3. Determine the optimum filler content that will have no significant reduction on the compressive strength of epoxy based polymer resin
4. Using the optimal mix design in activities 2 and 3, evaluate the six-month durability with respect to simulated water and saltwater environment of epoxy based polymer resin.
5. Analyse and interpret experimental results on the effects of elevated temperature and simulated environments on the physical and mechanical properties of epoxy based polymer resin.
6. Prepare and submit project dissertation.

AGREED:

Student Name: Ashlii Timms
16 / 04 / 2015

Supervisor Name: Allan Manalo
16 / 04 / 2015

Appendix B

Risk Assessment

APPENDIX B – RISK ASSESSMENT

TABLE 1RISK RATING

LIKELIHOOD	RISK RANKING MATRIX				
HIGH	M	H	E	E	E
SIGNIFICANT	M	H	H	E	E
MODERATE	L	M	H	H	H
LOW	L	L	M	M	M
NEGLIGIBLE	L	L	L	L	L
CONSEQUENCE	NEGLIGIBLE	LOW	MODERATE	MAJOR	CATASTROPHIC

TABLE 2 PROBABILITY

LIKELIHOOD DEFINITIONS	
A **high** likelihood	• It is expected to occur in most circumstances • There is a strong likelihood of the hazards reoccurring
A **significant** likelihood	• Similar hazards have been recorded on a regular basis • Considered that it is likely that the hazard could occur
A **moderate** likelihood	• Incidents or hazards have occurred infrequently in the past
A **low** likelihood	• Very few known incidents of occurrence • Has not occurred yet, but it could occur sometime
A **negligible** likelihood	• No known or recorded incidents of occurrence • Remote chance, may only occur in exceptional circumstance

TABLE 3 CONSEQUENCE

CONSEQUENCE DEFINITIONS	
Catastrophic	• Death of one or more people • Major environmental impact • Financial loss greater than $250,000
Major	• Extensive or multiple injuries (Hospitalisation required) • Minimal environmental impact • Major uncontained spills • Financial loss $100,000 - $250,000
Moderate	• Medical treatment required • Nil environmental impact • Large contained spills • Financial loss $50,000 - $99,999
Low	• First aid required • Small easily contained spills • Financial loss $5,000 - $49,999
Negligible	• No injuries • Minor delays • Little financial loss $0 - $4,999

TABLE 4 RECOMMENDED ACTION GUIDE

Abbrev.	Action Level	Descriptor
E	**Extreme**	The proposed task or process activity MUST NOT proceed until the supervisor has reviewd the task or process design and risk controls. They must take steps to firstly eliminate the risk and if this is not possible to introduce measures to control the risk by reducing the level of risk to the lowest level achievable. In the case of an existing hazard that is identified, controls must be put in place immediately.
H	**High**	Urgent action is required to eliminate or reduce the foreseeable risk arising from the task or process. The supervisor must be made aware of the hazard. However, the supervisor may give special permission for staff to undertake some high risk activities provided that system of work is clearly documented, specific training has been given in the required procedure and an adequate review of the task and risk controls has been undertaken. This includes providing risk controls identified in Legislation, Australian Standards, Codes of Practice. A detailed Standard Operating Procedure is required and monitoring of its implementation must occur to check the risk level
M	**Moderate**	Action to eliminate or reduce the risk is required within a specified period. The supervisor should approve all moderate risk task or process activities. A Standard Operating Procedure or Safe Work Method statement is required
L	**Low**	Manage by routine procedures

EVENT RISK ASSESSMENT

RISK ASSESSMENT TEMPATE

Date: 8 January 2015		**What is the task?**	**Prepare and test epoxy resin samples**
Faculty: Faculty of Health, Engineering and Sciences		**Location?**	**P9 and P11**
Assessor: Ashlii Timms		**What is operational significance?**	**Final year research project**
Hazards	**Risk rank**	**Control / Actions**	**Responsibility**
Chemical burn from Epoxy & hardener use	L	PPE: Gloves, overalls, safety glasses, mask, enclosed footwear. Enclosed room	Personal
Cut/physical injury or particles in eyes from de-moulding samples with hacksaw	L	PPE: Gloves, safety glasses Ensure samples are restrained when cutting mould	Personal
Physical injury while testing	L	Supervised and operated by an experienced staff member.	Personal
Burns from using oven to heat samples	L	PPE: Heat protection gloves	Personal

Appendix C

Raw Data for Study 1

TABLE C. 1 RESULTS FROM COMPRESSION TESTING

Specimen #	Diameter	Height	Area	Peak Load	Peak Stress	Specimen #	Comment
	mm	mm	mm^2	N	MPa		
1	29.71	28.56	693	33442	**48.24**	1	RT_0_1
2	29.53	29.07	685	34015	**49.67**	2	RT_0_2
3	29.35	29.24	677	31118	**45.99**	3	RT_0_3
4	29.71	30.08	693	41769	**60.25**	4	RT_20_1
5	29.58	28.12	687	36658	**53.34**	5	RT_20_2
6	29.55	28.64	686	36012	**52.51**	6	RT_20_3
7	29.57	31.61	687	33192	**48.33**	7	RT_40_1
8	29.6	32.76	688	28597	**41.56**	8	RT_40_2
9	29.54	31.04	685	31195	**45.52**	9	RT_40_3
10	29.66	29.16	691	29973	**43.38**	10	RT_60_1
11	29.68	31.88	692	24227	**35.02**	11	RT_60_2
12	29.83	32.09	699	22468	**32.15**	12	RT_60_3
13	29.59	28.25	688	7619	**11.08**	13	40_0_1
14	29.6	27.52	688	5059	**7.35**	14	40_0_2
15	29.6	27.52	688	8164	**11.86**	15	40_0_2
16	29.67	29.85	691	6272	**9.07**	16	40_0_3
17	29.78	28.01	697	10203	**14.65**	17	40_20_1
18	29.78	28.01	697	19825	**28.46**	18	40_20_1
19	29.59	28.25	688	22544	**32.78**	19	40_20_1
20	29.85	28.42	700	13710	**19.59**	20	40_20_2
21	29.85	28.42	700	24632	**35.2**	21	40_20_2
22	29.78	30.16	697	23590	**33.87**	22	40_20_3
23	29.69	32.84	692	14193	**20.5**	23	40_40_1
24	29.61	31.06	689	14252	**20.7**	24	40_40_2
25	29.56	30.04	686	15377	**22.41**	25	40_40_3
26	29.59	32.38	688	15844	**23.04**	26	40_60_1
27	29.66	31.4	691	18363	**26.58**	27	40_60_2
28	29.81	32.15	698	17786	**25.48**	28	40_60_3
29	29.51	30.94	684	2485	**3.63**	29	60_0_1
30	29.49	28.25	683	6068	**8.88**	30	60_0_2
31	29.6	26.98	688	2322	**3.37**	31	60_0_3
32	29.64	29.3	690	4340	**6.29**	32	60_20_1
33	29.58	28.66	687	4842	**7.05**	33	60_20_2
34	29.74	30.45	695	4102	**5.91**	34	60_20_3
35	29.69	31.19	692	5227	**7.55**	35	60_40_1
36	29.69	30.37	692	6330	**9.14**	36	60_40_2
37	29.63	32.08	690	4701	**6.82**	37	60_40_3

38	29.72	30.57	694	5667	**8.17**	38	60_60_1
39	29.64	31.56	690	6052	**8.77**	39	60_60_2
40	29.62	30.36	689	6746	**9.79**	40	60_60_3
41	29.51	29.71	684	3024	**4.42**	41	80_0_1
42	29.52	29.63	684	3044	**4.45**	42	80_0_2
43	29.51	29.61	684	2274	**3.33**	43	80_0_3
44	29.47	29.66	682	3502	**5.13**	44	80_20_1
45	29.64	30.49	690	2983	**4.32**	45	80_20_2
46	29.7	28.35	693	1710	**2.47**	46	80_20_3
47	29.77	31.75	696	3191	**4.58**	47	80_40_1
48	29.54	29.81	685	3135	**4.57**	48	80_40_2
49	29.67	29.57	691	2870	**4.15**	49	80_40_3
50	29.81	30.84	698	3973	**5.69**	50	80_60_1
51	29.69	31.42	692	3940	**5.69**	51	80_60_2
52	29.7	31.7	693	3465	**5**	52	80_60_3
53	29.5	29.5	683	6868	**10.05**	53	30_0_1
54	29.58	29.13	687	12918	**18.8**	54	30_0_2
55	29.57	28.35	687	30101	**43.83**	55	30_0_3
56	29.65	28.52	690	33682	**48.78**	56	30_20_1
57	29.62	29.4	689	29954	**43.47**	57	30_20_2
58	29.66	29.21	691	27762	**40.18**	58	30_20_3
59	29.72	30.77	694	25688	**37.03**	59	30_40_1
60	29.74	31.55	695	26544	**38.21**	60	30_40_2
61	29.7	31.97	693	26394	**38.1**	61	30_40_3
62	29.7	30.4	693	25505	**36.81**	62	30_60_1
63	29.6	31.26	688	25690	**37.33**	63	30_60_2
64	29.63	30.48	690	28959	**42**	64	30_60_3
Mean	**29.64**	**30**	**690**	**15878**	**23.01**		
Std Dev	**0.1**	**1.46**	**5**	**11974**	**17.37**		

🟨 **Data excluded from Analysis**

Appendix D

Raw Data for Study 2

D.1 MICROSCOPIC OBSERVATIONS

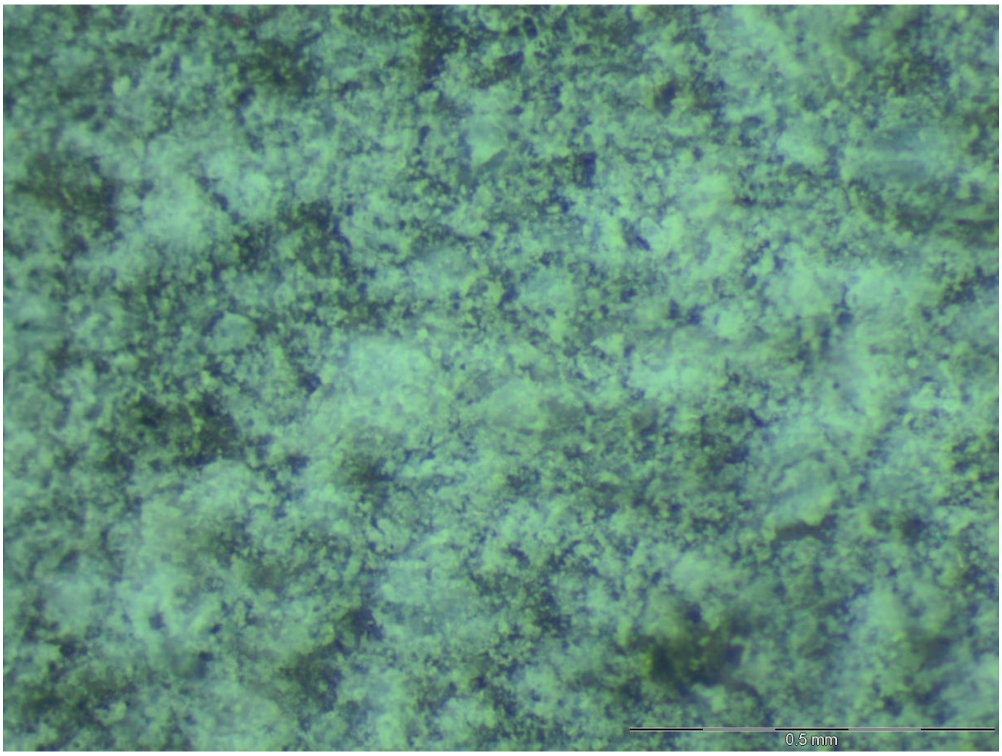

FIGURE D. 1 MICROSCOPIC OBSERVATION OF INITIAL SAMPLE

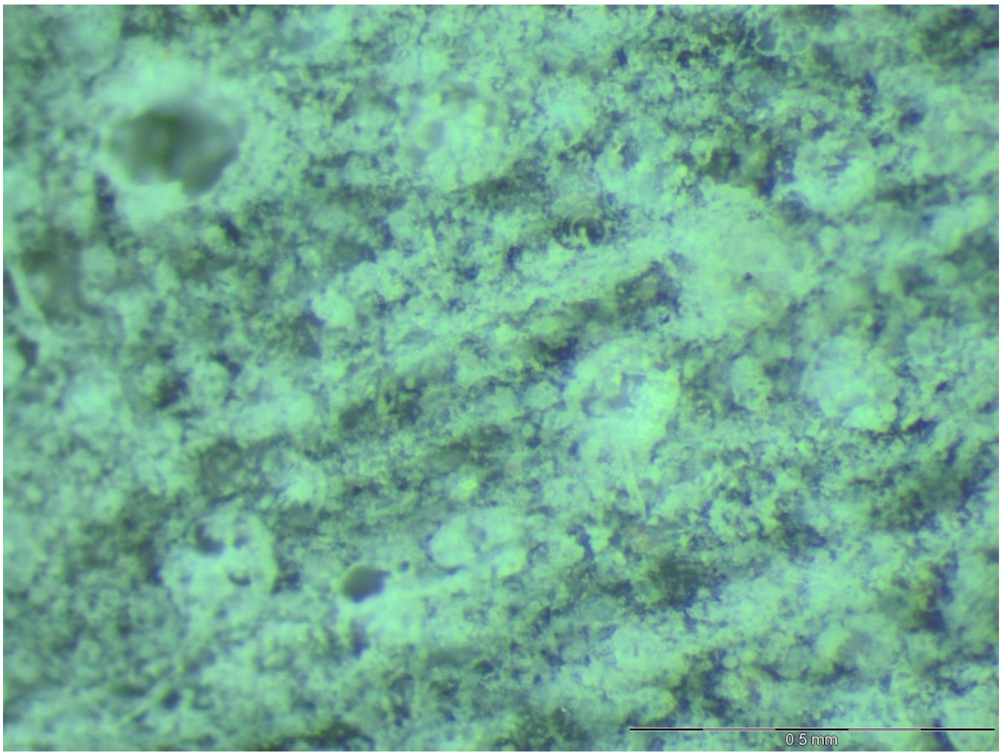

FIGURE D. 2 MICROSCOPIC OBSERVATION OF 1 DAY SAMPLE EXPOSED TO HYGROTHERMAL ENVIRONMENT

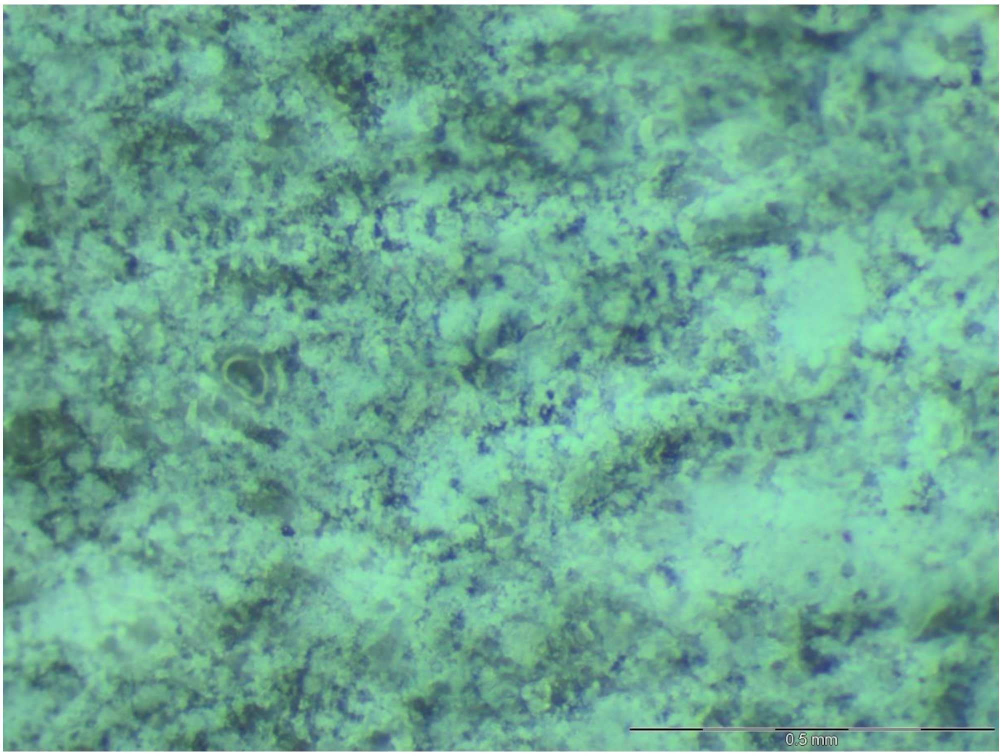

FIGURE D. 3 MICROSCOPIC OBSERVATION OF 3 DAY SAMPLE EXPOSED TO HYGROTHERMAL ENVIRONMENT

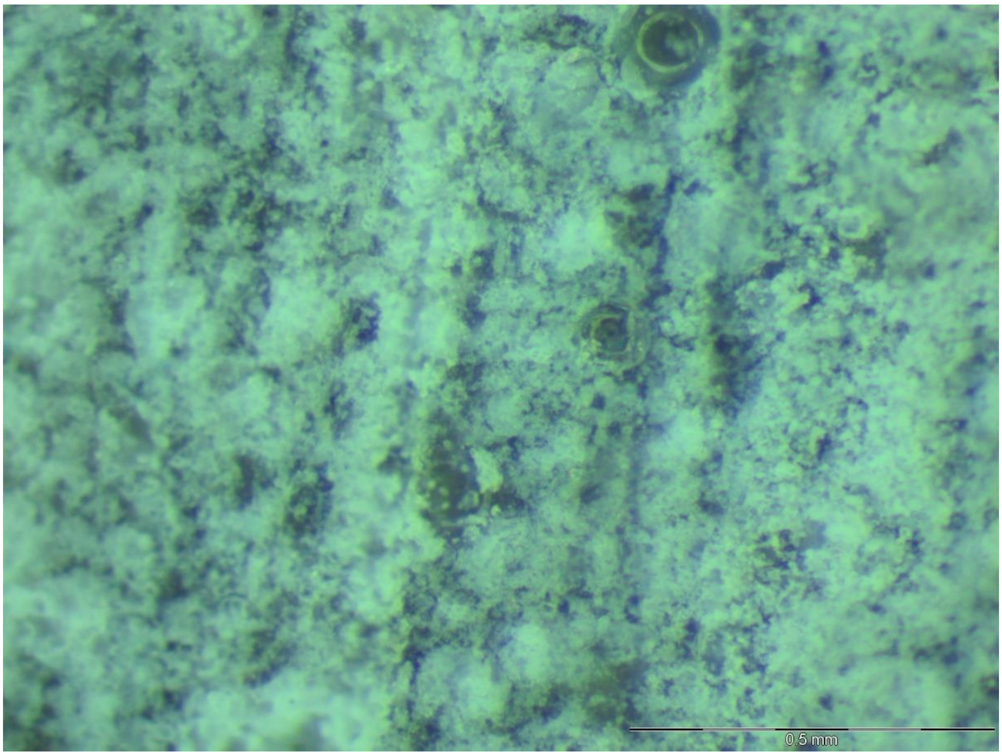

FIGURE D. 4 MICROSCOPIC OBSERVATION OF 7 DAY SAMPLE EXPOSED TO HYGROTHERMAL ENVIRONMENT

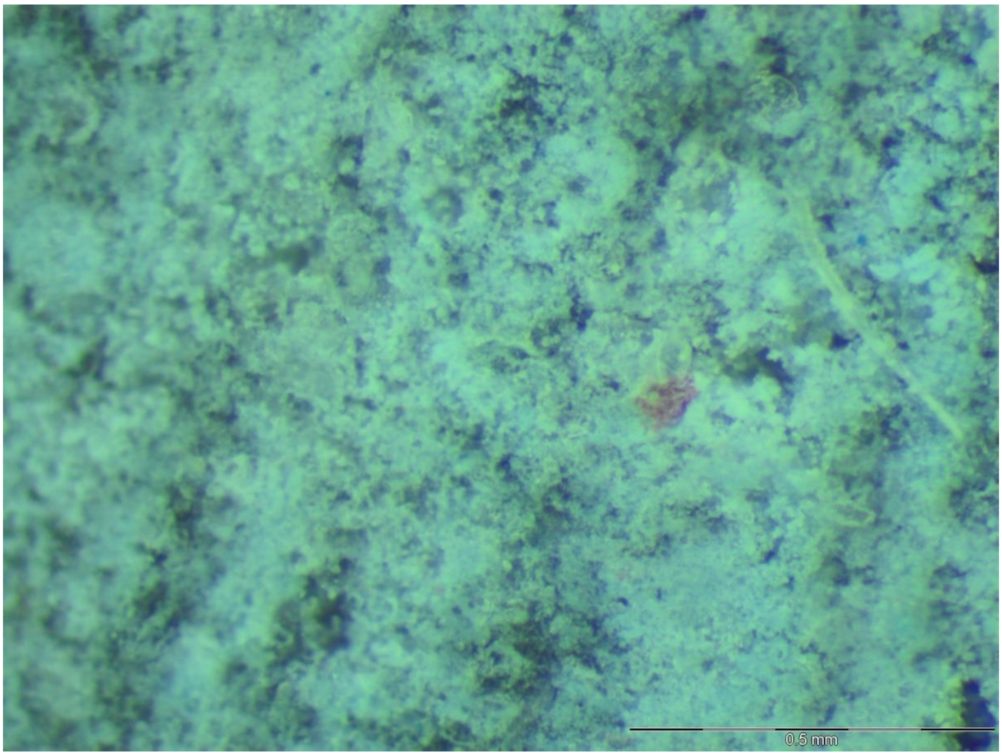

FIGURE D. 5 MICROSCOPIC OBSERVATION OF 28 DAY SAMPLE EXPOSED TO HYGROTHERMAL ENVIRONMENT

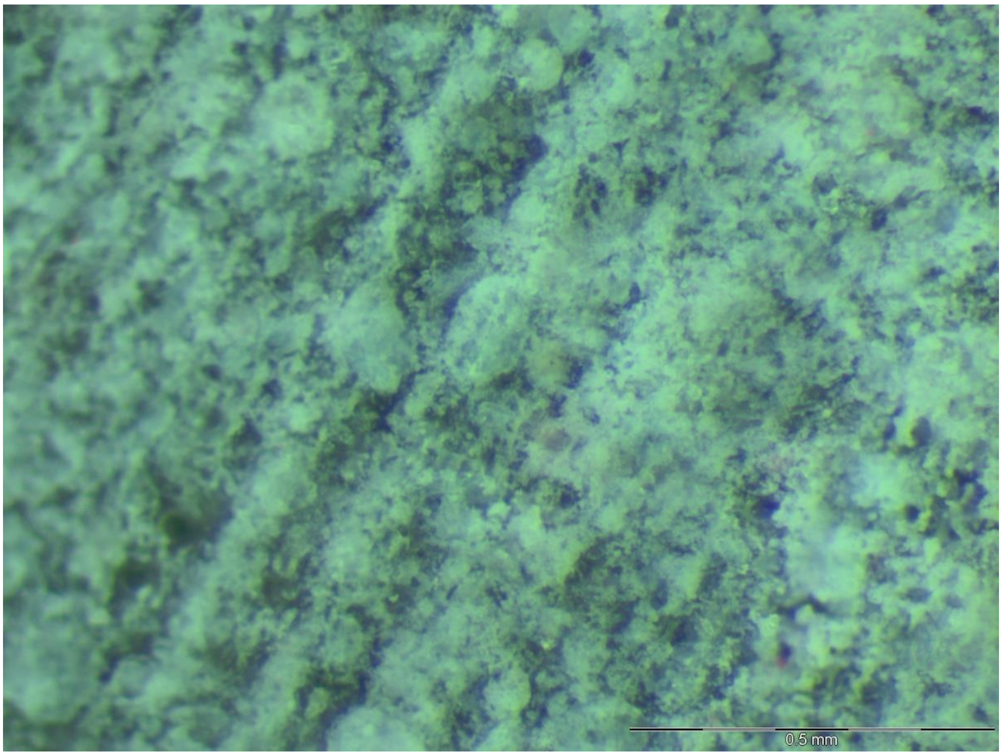

FIGURE D. 6 MICROSCOPIC OBSERVATION OF 7 DAY SAMPLE EXPOSED TO SALT-WATER ENVIRONMENT

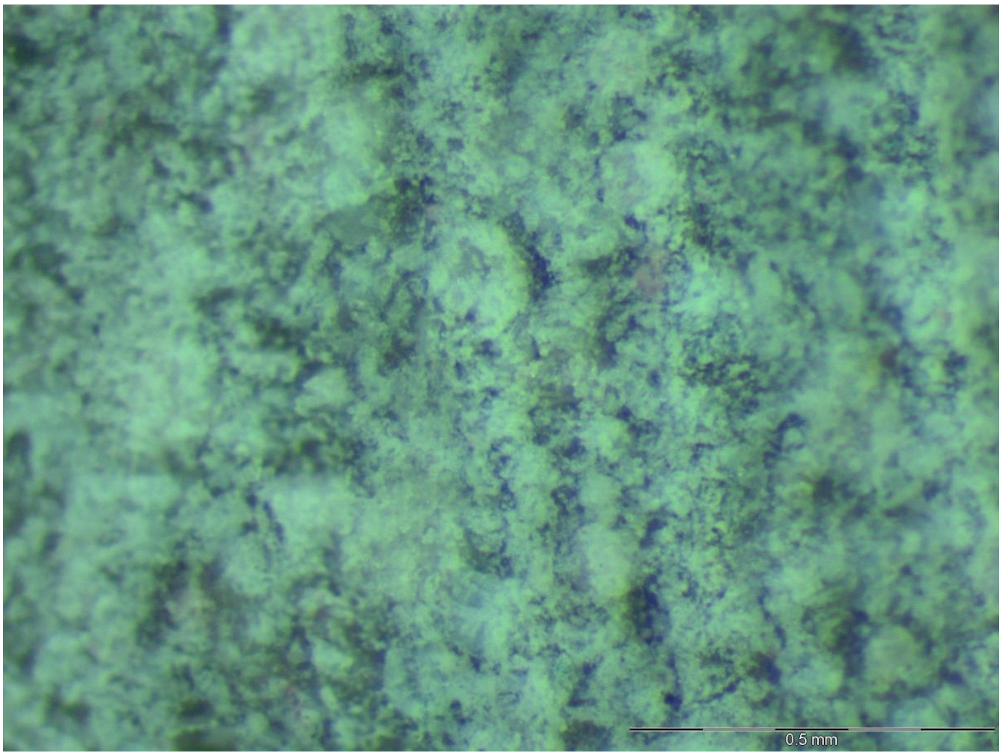

Figure D. 7 Microscopic Observation of 28 Day Sample Exposed to Salt-Water Environment

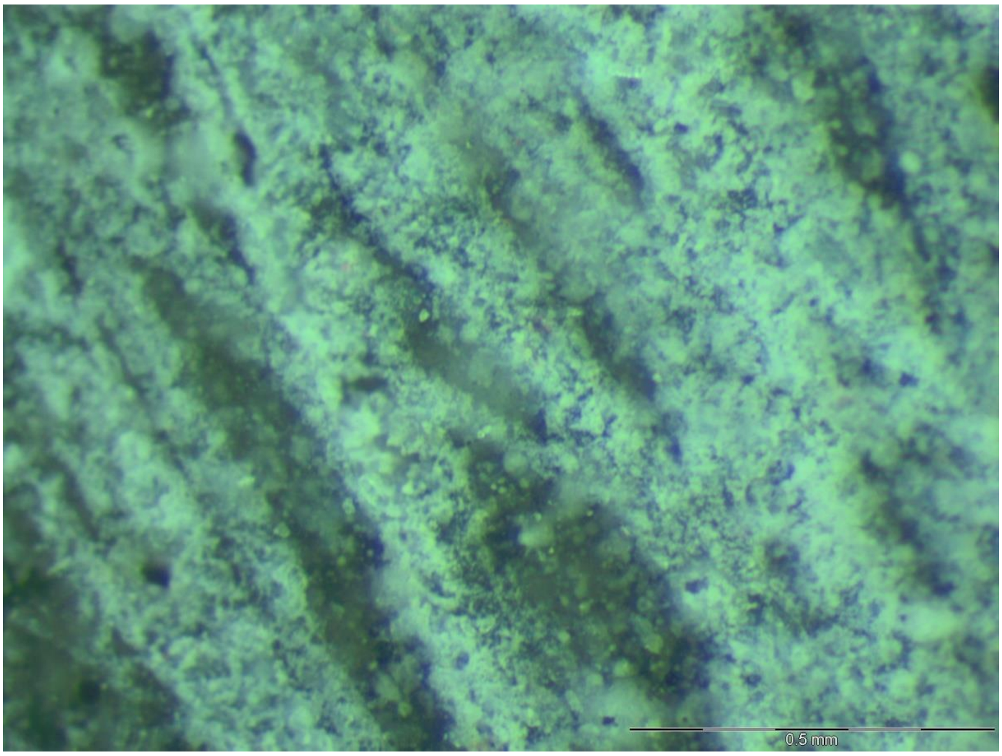

Figure D. 8 Microscopic Observation of 56 Day Sample Exposed to Salt-Water Environment

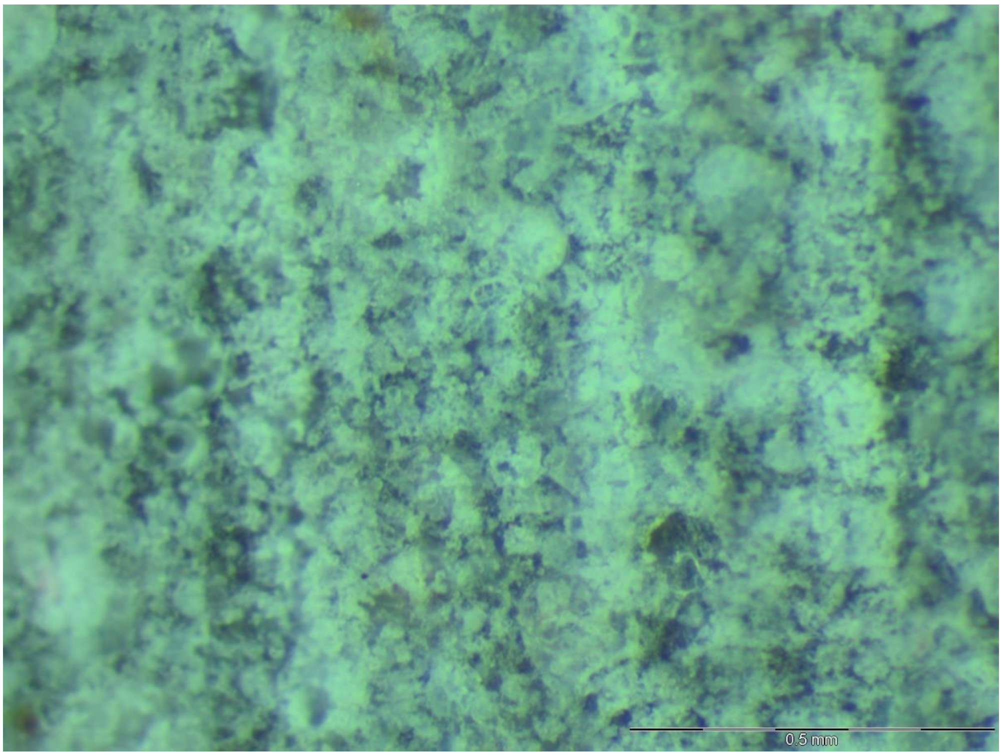

Figure D. 9 Microscopic Observation of 112 Day Sample Exposed to Salt-Water Environment

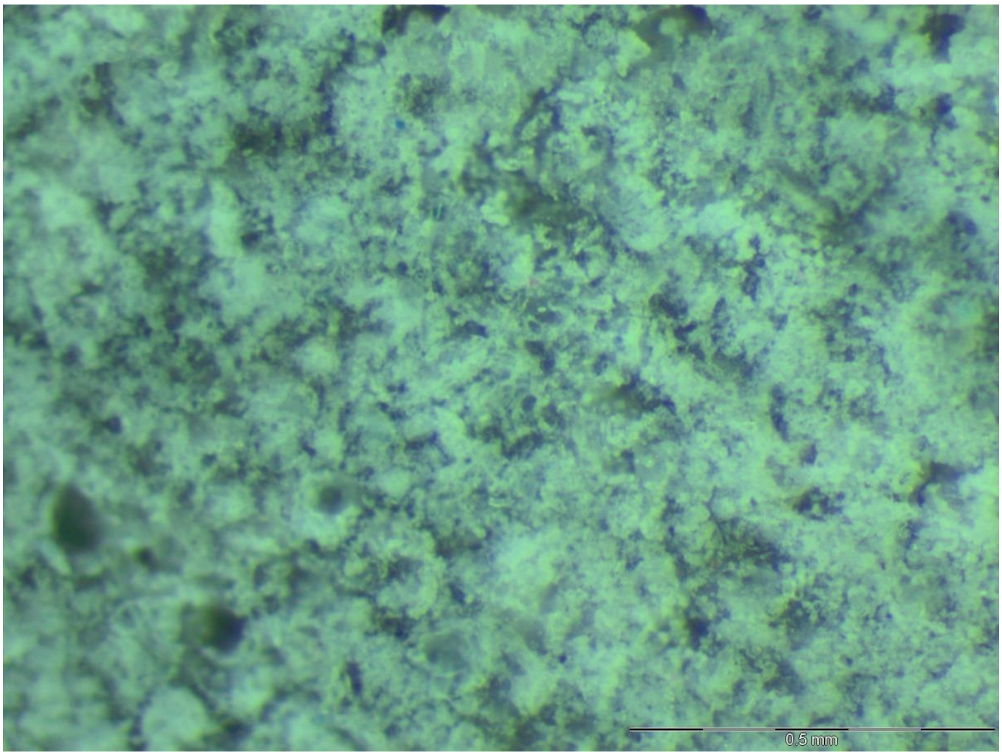

Figure D. 10 Microscopic Observation of 7 Day Sample Exposed to Water Environment

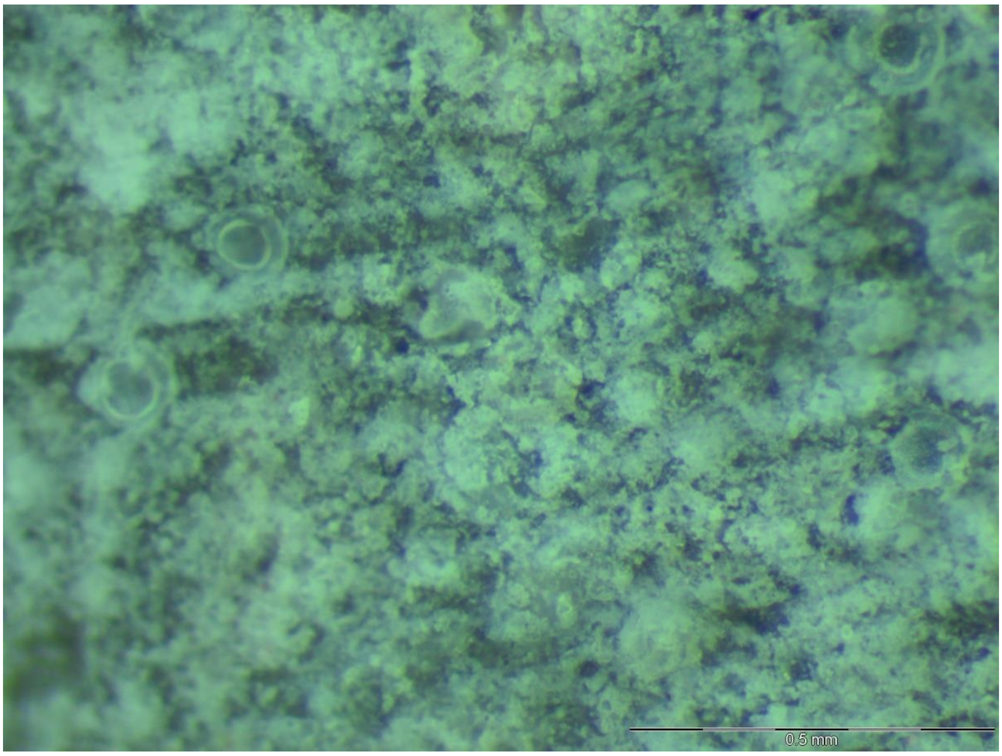

FIGURE D. 11 MICROSCOPIC OBSERVATION OF 28 DAY SAMPLE EXPOSED TO WATER ENVIRONMENT

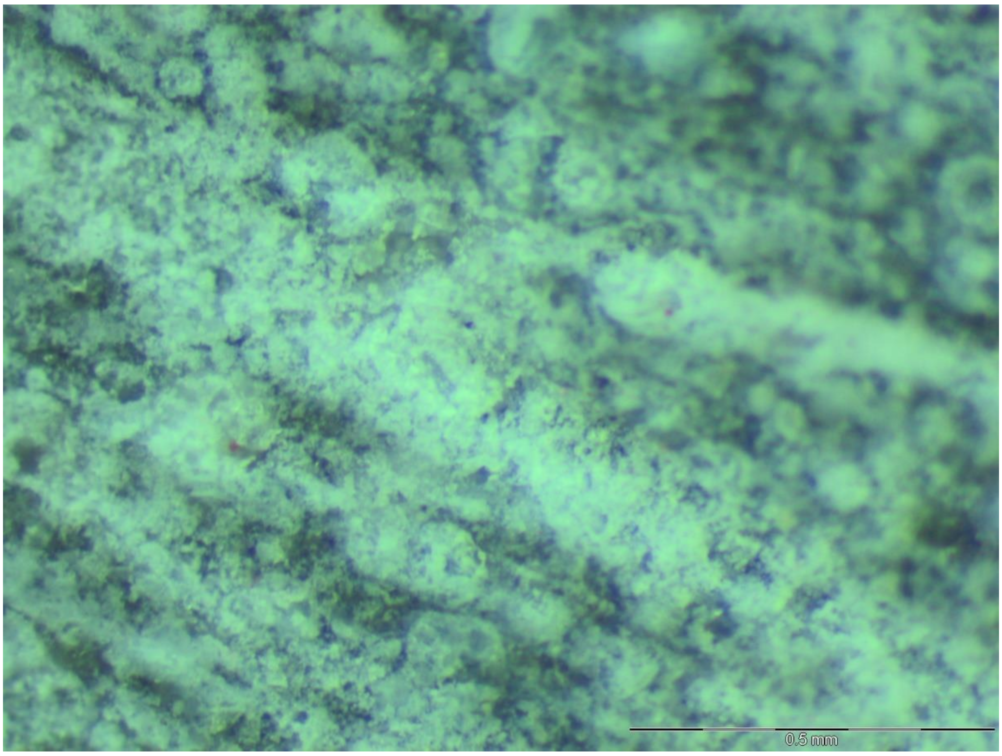

FIGURE D. 12 MICROSCOPIC OBSERVATION OF 56 DAY SAMPLE EXPOSED TO WATER ENVIRONMENT

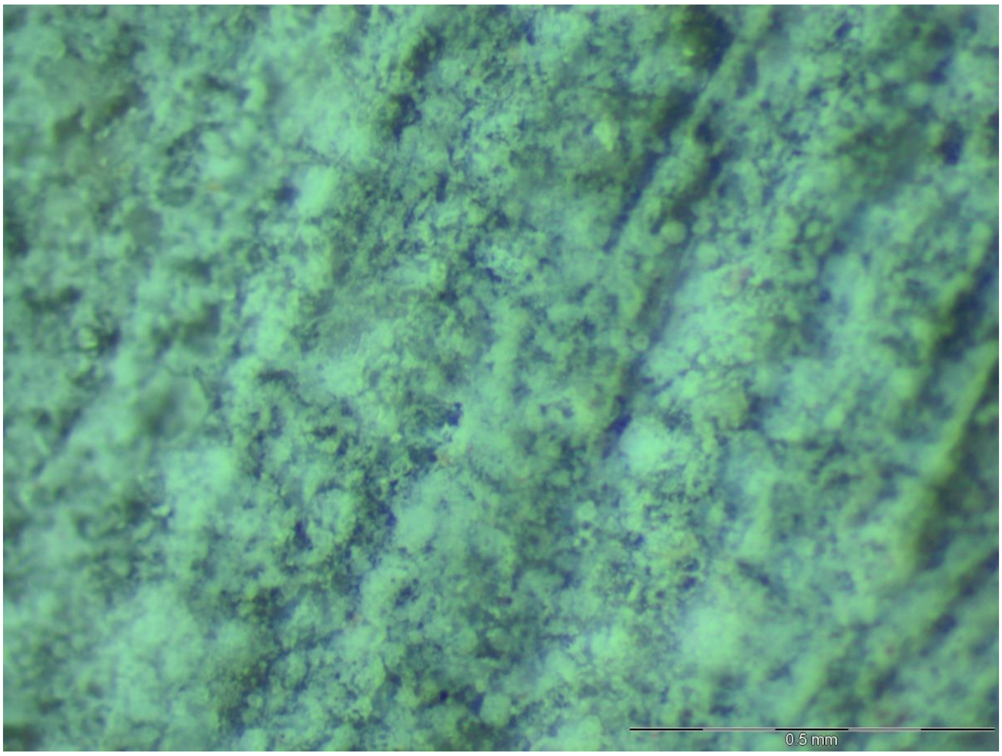

FIGURE D. 13 MICROSCOPIC OBSERVATION OF 112 DAY SAMPLE EXPOSED TO WATER ENVIRONMENT

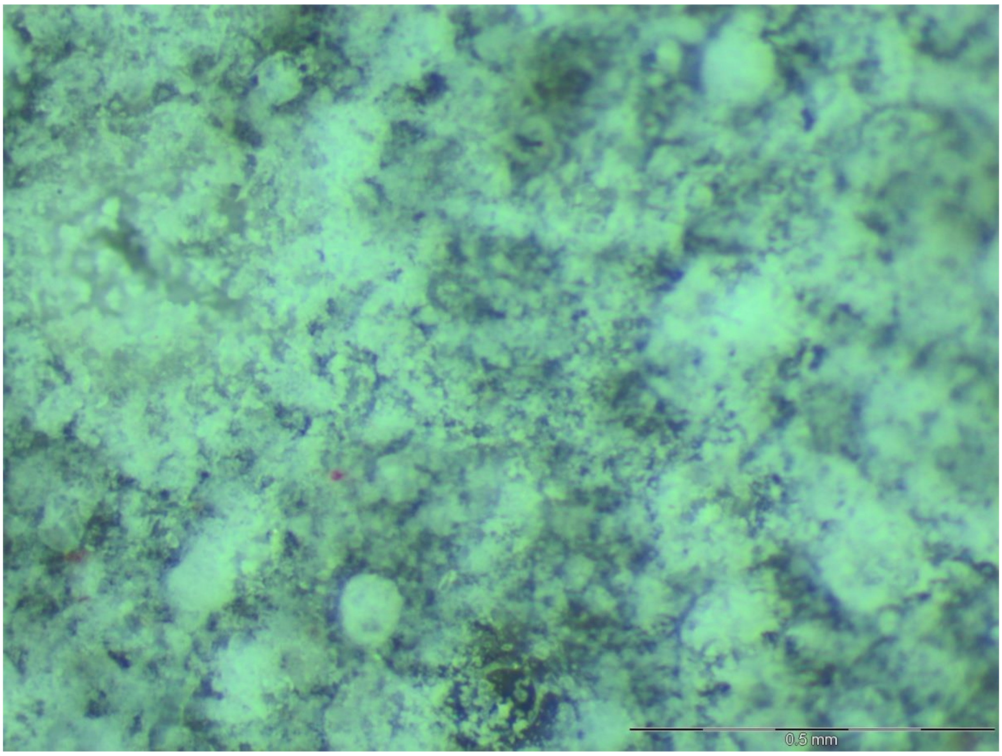

FIGURE D. 14 MICROSCOPIC OBSERVATION OF 56 DAY SAMPLE EXPOSED TO AIR ENVIRONMENT

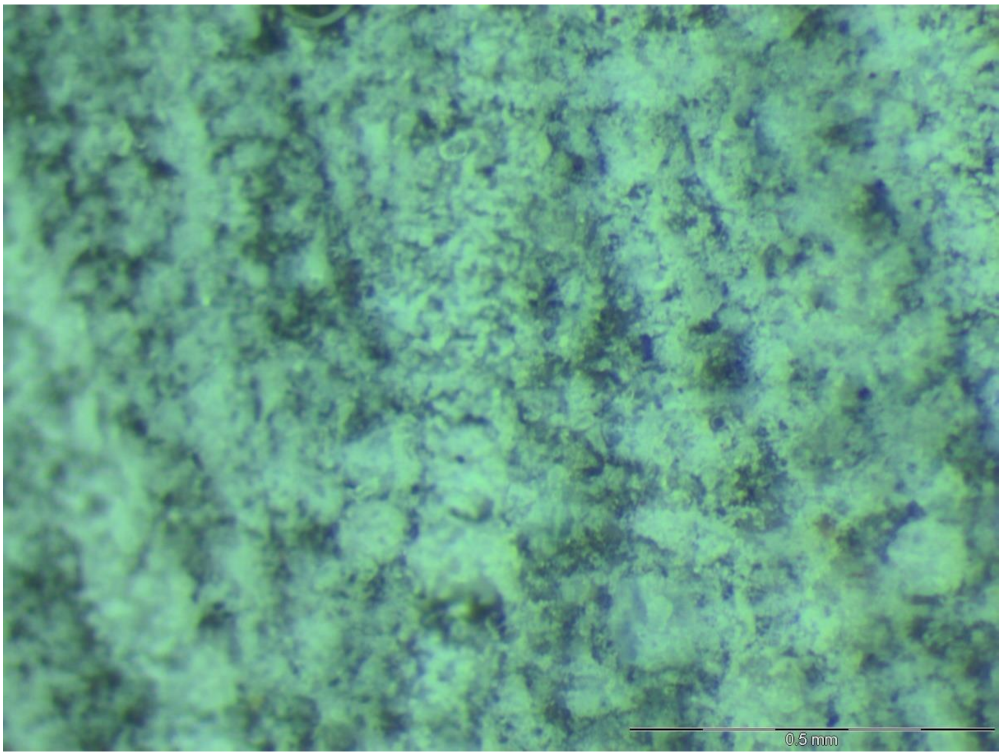

FIGURE D. 15 MICROSCOPIC OBSERVATION OF 112 DAY SAMPLE EXPOSED TO AIR ENVIRONMENT

D.2 RAW DATA

TABLE D. 1 RAW DATA FOR SPECIMENS EXPOSED TO AIR ENVIRONMENT

Collected Data

Initial

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa	
AI-1	29.68	30.81	28.6780	32.19	46.52	Control Samples
AI-2	29.60	31.14	29.2657	34.82	50.61	
AI-3	29.67	30.96	28.9970	35.27	51.02	
AI-4	29.70	31.93	29.8802	34.22	49.39	
AI-5	29.58	31.09	29.0996	36.14	52.59	
AI-6	29.53	31.25	29.1518			Untested
A7-1	29.65	30.48	29.3320			
A7-2	29.52	30.38	28.6705			
A7-3	29.58	30.83	28.9765			
A28-1	29.61	29.97	28.0719			
A28-2	29.68	32.26	30.1540			
A28-3	29.51	31.99	30.1430			
A56-1	29.50	30.62	28.7436			
A56-2	29.68	31.10	29.1813			
A56-3	29.55	32.22	30.0976			
A112-1	29.63	31.57	29.8356			
A112-2	29.57	30.96	28.9121			
A112-3	29.53	31.98	30.0655			
A168-1	29.60	31.12	29.3928			
A168-2	29.54	30.82	29.0025			
A168-3	29.54	31.50	29.5476			
A1Y-1	29.54	29.81	27.6333			
A1Y-2	29.61	28.57	26.7567			
A1Y-3	29.45	30.99	28.8395			
A2Y-1	29.52	29.52	27.8085			
A2Y-2	29.61	31.25	29.3463			
A2Y-3	29.61	30.36	28.7304			

1 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)
A7-1	29.69	30.83	29.3316
A7-2	29.71	30.37	28.6733

A7-3	29.76	30.87	28.9764
A28-1	29.57	30.08	28.0713
A28-2	29.76	32.29	30.1539
A28-3	29.56	31.78	30.1437

3 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)
A7-1	29.69	30.96	29.3322
A7-2	29.58	30.38	28.6733
A7-3	29.64	30.98	28.9765
A28-1	29.60	30.06	28.0713
A28-2	29.70	32.32	30.1541
A28-3	29.62	31.78	30.1434

7 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
A7-1	29.65	30.58	29.3333	36.19	52.42
A7-2	29.52	30.28	28.6731	36.93	53.96
A7-3	29.48	30.69	28.9761	37.18	54.47
A28-1	29.54	29.93	28.0706		
A28-2	29.70	32.23	30.1559		
A28-3	29.51	31.67	30.1430		

Failure

28 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
A28-1	29.57	29.85	28.0800	41.48	60.40
A28-2	29.70	32.23	30.1618	41.33	59.66
A28-3	29.54	31.61	30.1506	41.30	60.26
A56-1	29.53	30.65	28.7510		
A56-2	29.70	31.03	29.1918		
A56-3	29.62	32.20	30.1075		

56 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
A56-1	29.56	30.69	28.7549	44.10	64.25
A56-2	29.73	30.98	29.1967	42.81	61.67
A56-3	29.62	32.17	30.1108	41.82	60.69

A112-1	29.59	31.47	29.8463
A112-2	29.57	30.83	28.9241
A112-3	29.52	31.90	30.0778

112 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
A112-1	29.60	31.48	29.8417	46.06	66.98
A112-2	29.60	30.98	28.9215	40.48	58.82
A112-3	29.57	32.00	30.0752	45.73	66.59
A168-1	29.69	31.14	29.4031		
A168-2	29.60	30.82	29.0126		
A168-3	29.71	31.58	29.5592		

168 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
A168-1	29.62	30.92	29.4016	47.91	69.52
A168-2	29.45	30.62	29.0118	46.45	68.20
A168-3	29.57	31.38	29.5579	46.46	67.65
A1Y-1	29.52	29.81	27.6448		
A1Y-2	29.54	28.51	26.7668		
A1Y-3	29.36	30.54	28.8516		

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Report

TABLE D. 2 RAW DATA FOR SPECIMENS EXPOSED TO WATER ENVIRONMENT

Collected Data

Initial			
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**
W7-1	29.66	31.29	29.4137
W7-2	29.53	31.57	29.4845
W7-3	29.54	30.38	28.2428
W28-1	29.61	30.00	28.2480
W28-2	29.43	30.08	28.5591
W28-3	29.49	30.53	28.5387
W56-1	29.59	29.20	27.4413
W56-2	29.65	31.12	29.1729
W56-3	29.57	30.74	28.7842
W112-1	29.47	30.34	28.4467
W112-2	29.52	31.77	29.7451

W112-3	29.66	31.75	29.6485
W168-1	29.47	31.51	29.4762
W168-2	29.53	29.90	28.1653
W168-3	29.63	28.79	27.0078
W1Y-1	29.54	30.46	28.6503
W1Y-2	29.60	30.74	28.8860
W1Y-3	29.55	30.78	28.4960
W2Y-1	29.27	29.43	27.4519
W2Y-2	29.63	31.48	29.4070
W2Y-3	29.56	30.76	28.5936

1 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)
W7-1	29.71	31.30	29.4320
W7-2	29.58	31.31	29.5022
W7-3	29.58	30.23	28.2615
W28-1	29.64	30.03	28.2649
W28-2	29.51	30.11	28.5768
W28-3	29.48	30.47	28.5574

3 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)
W7-1	29.64	31.07	29.4499
W7-2	29.59	31.13	29.5154
W7-3	29.57	30.14	28.2817
W28-1	29.61	29.93	28.2790
W28-2	29.43	29.85	28.5941
W28-3	29.47	30.27	28.5712

7 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
W7-1	29.64	31.17	29.4567	38.59	55.93
W7-2	29.52	31.18	29.5257	38.81	52.32
W7-3	29.51	30.04	28.2925	36.14	52.84
W28-1	29.64	29.84	28.2904		
W28-2	29.39	29.74	28.5965		
W28-3	29.46	30.19	28.5804		

28 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
W28-1	29.62	29.95	28.3058	42.48	61.64
W28-2	29.38	29.74	28.6150	41.40	61.07
W28-3	29.47	30.31	28.5960	39.39	57.75
W56-1	29.61	28.98	27.4984		
W56-2	29.61	30.97	29.2370		
W56-3	29.54	30.67	28.8405		

56 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
W56-1	29.56	29.01	27.5002	40.26	58.67
W56-2	29.63	30.99	29.2392	42.70	61.93
W56-3	29.56	30.66	28.8460	42.20	61.50
W112-1	29.59	30.18	28.5144		
W112-2	29.50	31.64	29.8130		
W112-3	29.65	31.59	29.8169		

112 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
W112-1	29.47	30.12	28.5312	42.76	62.68
W112-2	29.52	31.53	29.8401	43.81	64.00
W112-3	29.60	31.38	29.8397	41.17	59.83
W168-1	29.45	31.31	29.5690		
W168-2	29.46	29.58	28.2521		
W168-3	29.58	28.25	27.0950		

168 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)	Peak Load kN	Peak Stress Mpa
W168-1	29.44	31.33	29.5784	42.15	61.92
W168-2	29.45	29.29	28.2560	43.35	63.65
W168-3	29.56	28.26	27.0976	41.83	60.95
W1Y-1	29.55	30.02	28.7525		
W1Y-2	29.62	30.09	28.9788		
W1Y-3	29.48	30.39	28.5823		

Past the Scope of my Report

TABLE D. 3 RAW DATA FOR SPECIMENS EXPOSED TO SALT-WATER ENVIRONMENT

Collected Data

Initial

Sample	Diameter (mm)	Height (mm)	Weight (g)
S7-1	29.59	30.99	29.0933
S7-2	29.44	30.60	28.5640
S7-3	29.48	30.50	28.5427
S28-1	29.65	31.55	29.7185
S28-2	29.61	30.07	28.2677
S28-3	29.52	31.13	29.2808
S56-1	29.50	32.07	30.1204
S56-2	29.51	28.94	27.3542
S56-3	29.55	28.98	27.2194
S112-1	29.45	30.75	28.6715
S112-2	29.63	30.60	29.2310
S112-3	29.44	31.78	29.7433
S168-1	29.60	31.29	29.3814
S168-2	29.62	30.70	28.7870
S168-3	29.61	31.21	29.1945
S1Y-1	29.49	30.62	28.7968
S1Y-2	29.53	30.14	28.1107
S1Y-3	29.54	30.05	28.1187
S2Y-1	29.61	30.88	29.0390
S2Y-2	29.65	30.07	27.9920
S2Y-3	29.67	31.56	29.7310

1 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)
S7-1	29.69	30.73	29.1061
S7-2	29.43	30.77	28.5746
S7-3	29.52	30.45	28.5535
S28-1	29.71	31.47	29.7305
S28-2	29.63	30.09	28.2783
S28-3	29.65	30.95	29.2925

3 DAY

Sample	Diameter (mm)	Height (mm)	Weight (g)
S7-1	29.66	30.35	29.1278

S7-2	29.51	30.57	28.5941
S7-3	29.57	30.23	28.5722
S28-1	29.70	31.45	29.7528
S28-2	29.64	30.01	28.2923
S28-3	29.55	30.96	29.3086

7 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
S7-1	29.54	30.52	29.1290	35.86	52.32
S7-2	29.40	30.43	28.6000	36.34	53.53
S7-3	29.48	30.18	28.5788	37.68	55.21
S28-1	29.69	31.41	29.7560		
S28-2	29.56	29.96	28.3014		
S28-3	29.50	30.81	29.3164		

28 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
S28-1	29.68	31.35	29.7825	41.34	59.75
S28-2	29.61	30.03	28.3180	40.84	59.31
S28-3	29.57	30.95	29.3363	41.58	60.55
S56-1	29.52	31.89	30.1820		
S56-2	29.54	28.64	27.2795		
S56-3	29.53	28.77	27.2795		

56 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
S56-1	29.52	31.97	30.1832	42.51	62.11
S56-2	29.49	28.65	27.4135	42.67	62.47
S56-3	29.50	28.72	27.2821	42.00	61.44
S112-1	29.43	30.52	28.7365		
S112-2	29.62	30.11	29.2906		
S112-3	29.40	31.54	29.8116		

112 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
S112-1	29.42	30.45	28.7611	43.14	63.46
S112-2	29.59	30.00	29.3106	42.98	62.51
S112-3	29.44	31.44	29.8272	42.31	62.15
S168-1	29.55	30.99	29.4680		

S168-2	29.57	30.12	28.8706
S168-3	29.57	30.55	29.2825

168 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
S168-1	29.56	31.01	29.4755	43.65	63.61
S168-2	29.56	30.07	28.8800	42.41	61.80
S168-3	29.54	30.45	29.2858	41.37	60.37
S1Y-1	29.43	30.30	28.8920		
S1Y-2	29.51	30.09	28.2012		
S1Y-3	29.47	29.80	28.2033		

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TABLE D. 4 RAW DATA FOR SPECIMENS EXPOSED TO HYGROTHERMAL ENVIRONMENT

Collected Data

Initial			
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**
H1-1	29.51	31.77	29.8281
H1-2	29.59	31.79	29.8702
H1-3	29.49	29.28	27.3515
H3-1	29.67	30.47	28.536
H3-2	29.47	30.63	28.6671
H3-3	29.44	30.25	28.3018
H7-1	29.45	30.38	28.3678
H7-2	29.45	31.05	28.9562
H7-3	29.59	30.78	28.9812
H28-1	29.49	31.24	28.9581
H28-2	29.66	30.96	29.0682
H28-3	29.53	30.81	28.5859

2 HRS			
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**
H1-1	29.50	31.61	29.8496
H1-2	29.67	31.43	29.8980
H1-3	29.55	29.15	27.3710
H3-1	29.67	30.24	28.5490
H3-2	29.57	30.26	28.6814
H3-3	29.51	30.06	28.3156

4 HRS			
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**
H1-1	29.38	31.53	29.8397
H1-2	29.51	31.41	29.8865
H1-3	29.40	29.12	27.3699
H3-1	29.51	30.21	28.5489
H3-2	29.45	30.15	28.6820
H3-3	29.35	29.97	28.3170

6 HRS			
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**
H1-1	29.24	31.44	29.8374
H1-2	29.32	31.38	29.8824
H1-3	29.19	29.10	27.3683
H3-1	29.38	30.18	28.5483
H3-2	29.28	30.08	28.6825
H3-3	29.18	29.82	28.3182

1 DAY					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
H1-1	29.58	31.81	29.8392	33.91	49.35
H1-2	29.73	31.75	29.8883	34.56	49.78
H1-3	29.56	29.39	27.3708	34.62	50.44
H3-1	29.72	30.53	28.5527		
H3-2	29.52	30.80	28.6832		
H3-3	29.54	30.32	28.3231		

3 DAYS					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
H3-1	29.71	30.41	28.5790	39.05	56.33
H3-2	29.53	30.51	28.7090	38.36	56.00
H3-3	29.51	30.27	28.3527	39.27	57.42
H7-1	29.47	30.45	28.4208		
H7-2	29.48	31.17	29.0098		
H7-3	29.68	30.69	29.0304		

7 DAYS					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**

H7-1	29.45	30.17	28.4330	40.75	59.83
H7-2	29.41	30.86	29.0172	40.85	60.14
H7-3	29.67	30.61	29.0370	41.28	59.71
H28-1	29.48	31.14	29.0208		
H28-2	29.64	30.72	29.1306		
H28-3	29.54	30.48	28.6418		

Failure

28 DAYS[1]					
Sample	**Diameter (mm)**	**Height (mm)**	**Weight (g)**	**Peak Load kN**	**Peak Stress Mpa**
H28-1	29.56	31.06	29.0083	42.93	62.55
H28-2	29.70	30.63	29.1260	43.90	63.37
H28-3	29.57	30.28	28.6375	40.87	59.52

Notes	
1	The samples were removed before 28 days from the hot pot