

SHEAR PROPERTY OF STRUCTURAL EPOXY ADHESIVE EXPOSED TO TROPICAL WEATHERING CONDITION

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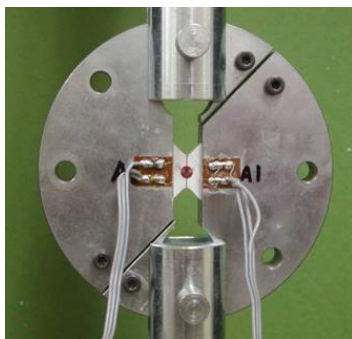
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Graphical abstract



Abstract

As a consequence of tropical climate featuring abundant rain and sunshine throughout the year, adhesive bonded joints undergo substantial exposure to moisture and elevated temperatures. It is known that the degradation of adhesive materials such as structural epoxy due to weathering could affect the overall bond performance of structural integrity of reinforced concrete such as carbon fibre reinforced polymer composites (CFRP) plate system. The objective of the study is to investigate the in-plane shear properties of structural epoxy material exposed to tropical environmental conditions using Arcan Test Method. The epoxy adhesive was casted in a closed metal mould to produce butterfly shaped specimens. The specimens were exposed to four conditions; laboratory, outdoor, plain water, and salt water. The specimens were tested for shear properties and failed in brittle form. Microstructure analysis was performed to study the fracture surface of the test specimens. The study showed that the tropical exposure conditions influenced the shear strength of the epoxy material, especially for those exposed to plain and salt water conditions, which were 32% lower than the control specimen for specimens exposed to plain water followed by salt water (26.6%), laboratory (25.4%) and finally outdoor (18.4%).

Keywords: Shear test, arcan test method, structural epoxy, tropical weathering, microstructure analysis.

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1.0 INTRODUCTION

Mechanical properties are measured by which loads or boundary displacements are applied in such a way that the relation between stress and strain at a typical point can be inferred. In brittle materials, the condition is further complicated by the tendency of fracture through the initiation and propagation of cracks at different locations throughout the material [1]. The shear properties of the brittle epoxy adhesive is one aspect that the user tends to neglect as it is only a small part. However, it is crucial in designing externally bonded fibre reinforced polymer composites (FRP) to reinforce concrete members. The selection of adhesive is important to provide sufficient material integrity in order to produce uniform stress throughout

the bond area. This is necessary to avoid premature failure such as adherent failure, cleavage and interfacial failure of the adhesive.

A study conducted by Karbhari and Zhao [2] had shown that the effect of exposure conditions on composite and composite-concrete interfaces was degradation due to moisture uptake. Their study involved the application of GFRP and CFRP composites that were externally bonded to the tension face of concrete beam specimens. The specimens were exposed to fresh water, salt water, freeze/thaw cycles and below freezing temperatures. The flexural load test results indicated that the degradation occurred primarily at the interface level of FRP-concrete and FRP itself due to changes in composite stiffness caused by resin plasticization. They also

discovered that the moisture absorption rate was higher in exposure to fresh water compared to sea water.

On the other hand, not much research has been conducted on the long-term performance of reinforced concrete beams strengthened using FRP plate-bonded system exposed to natural weather. Thus, this area needs further investigation especially with different exposure conditions. In this regard, the long-term durability of the FRP-plate bonded system exposed to different aggressive environments needs to be addressed especially for the exposure to tropical climate in which at present the data are very limited. In the plate bonding system the penetration of moisture may also occur through the resin via micro cracks, which can lead to local debonding of the FRP plate. Furthermore, most of the FRP reinforcements that have been developed in temperate countries were tested for durability under conditions simulating those countries. Since the tropical climate experiences abundant rain and sunshine throughout the year, it would be essential to assess the long-term durability of the FRP plate-bonded system in this Southeast Asian region [3].

Arcan *et al.* [4] introduced a new method of testing material shear properties under uniform plane stress conditions by means of a specially designed plane specimen. The fixture was used to determine the shear properties for various materials such as polymer composite, sandwich materials system, metals, human bones and solid polymers [5-15]. Their photoelasticity result had shown that in the significant section of the specimen it was possible to produce a uniform plane stress with high degree of accuracy. The compact nature of the Arcan fixture offered an advantage to obtain the shear properties in all in-plane directions in a relatively simple manner. The Arcan fixture was used to produce both shear and axial forces to the test specimen and this special case of loading produced pure shear on the significant section and the experimental results were encouraging and acceptable with high degree of confidence.

The present study focuses on the use of Arcan test method to determine the shear strength and modulus of structural epoxy adhesive exposed to tropical weathering conditions

2.0 MATERIALS AND METHODS

Epoxy resin namely SelfixCarbofibe adhesive, supplied by Exchem, United Kingdom was used. The properties of SelfixCarbofibe adhesive were produced by blending/mixing a modified epoxy resin and inorganic fillers to form a base component, which was chemically activated by a thixotropic formulated amine hardener. The mixing ratio and detailed procedures were performed according to supplier data sheet. The epoxy was left to cure for 24 hours at ambient laboratory condition.

The specimen geometry is as shown in Figure 1 was prepared. The 90° notches were formed at the centre

of 60 mm length (at the top and bottom) such that the distance between notches left about 10 mm at the middle, known as specification section, to introduce shear field. A notch radius of 1.5 mm was produced to minimise stress concentration and also to produce a uniform shear stress distribution along the significant section [14].

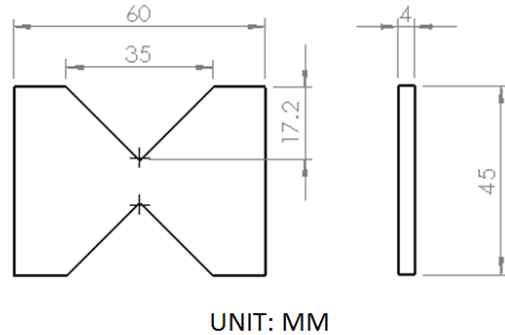


Figure 1 Arcan butterfly shaped specimen geometry

2.1 Specimen Preparation

The butterfly shaped cast epoxy specimens were prepared by mixing epoxy and hardener with the ratio of 3:1 (Figure 2). The butterfly shaped specimens were cast using a female mould (Figure 3). Five pieces of butterfly shaped specimens were prepared in each casting.



Figure 2 A mixing process using electric mixer

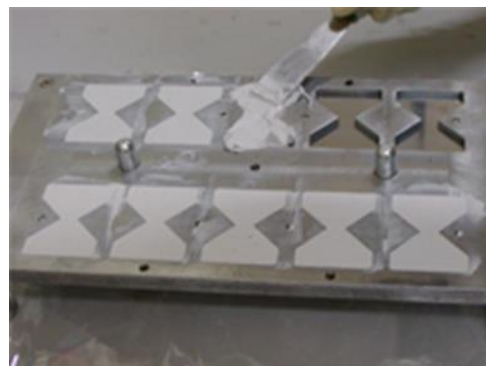


Figure 3 Filling the epoxy mixture into the mould cavity

A metal block weight of about 10 kg was placed onto the top of the mould to produce an extra uniform pressure on the mould. Finally the specimens were left in a laboratory environmental condition with temperatures ranging from 23 to 33 °C for at least 24 hours for curing prior to demoulding and polishing as shown in Figures 4 and 5.



Figure 4 Specimen demoulding using Teflon block.



Figure 5 Polishing

2.2 Durability Experimental Programme

Table 1 summarised, the specimens exposure to four different environmental conditions that reflects tropical climatic conditions, namely; laboratory condition (LB), outdoor condition (OD), exposure to plain water (PW) and salt water (SW) under wet/dry cycles. The wet and dry cycles were 7 days wetting and 7 days drying.

For the laboratory (LB) exposure condition, the specimens were exposed to a condition of 75 to 90% relative humidity (RH) and 23 to 33°C room temperatures. The specimens were placed on aluminium angle bars supported by mild steel rack and held in a horizontal position. The specimens exposed surfaces were rotated weekly during the exposure period of 6 months. Plain water and salt water was selected in order to study the effects of prolonged immersion of specimens in tropical climate and ocean

tropical water. Potable water was used for water resistance exposure condition.

Table 1 Sample codes for every exposure condition

| Exposure conditions | Sample code |
|--|-------------|
| Control | ESST-CO |
| Laboratory | ESLT-LB |
| Outdoor | ESLT-OD |
| Salt water (24 cycles) Exposure for 6 months 1 week/cycle | ESLT-SW |
| Plain water (24 cycles wet/dry) Exposure for 6 months 1 week/cycle | ESLT-PW |

Immersion of specimens in plain water and salt water was done by using 50 litre capacity tanks. The specimens were tested within 2 weeks of exposure time. The tanks were filled with plain (tap) water and salt at 1: 27.5 ratio. For outdoor exposure conditions the specimens were placed on aluminium bars. The specimens' surfaces were alternately rotated (weekly). At the end of the exposure period, the specimens were brought into the laboratory for the preparation of final load test to be conducted within two weeks.

2.3 Arcan Test Set-up

The modified Arcan test fixture used in this study consisted of a pair of parts A and B as shown in Figure 6. The exact shape and size of specimen was mounted into part A followed by part B. Both parts were tightened by screws to ensure that the specimen was tightly gripped between the fixtures to prevent slippage and misalignment during loading. The complete assembly of the fixtures was attached to the holder at the lower and upper parts accordingly prior to attachment to the Instron Universal Testing Machine.

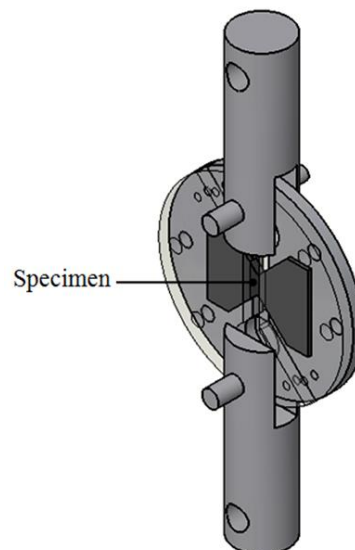


Figure 6 The specimen's position inside the Arcan test fixture

The tensile loads were applied on the Arcan fixture through holders before the loading configuration was changed to shear mode. The direction of the principal shear then acted in the direction of $\pm 45^\circ$ in relation to the specimen horizontal axis. The fixture holders were attached to the loading machine, namely Universal Testing Machine by two pins with a diameter of 15 mm. The complete Arcan grip fixture consisting of the butterfly specimen was connected to the holders by using two pins at the upper and lower sections of the Arcan plate. All the pins were carefully checked to ensure that the connection was in perfect adjustment before applying the load as displayed in Figures 7 and 8.



Figure 7 Attachment of grip to holder by pin

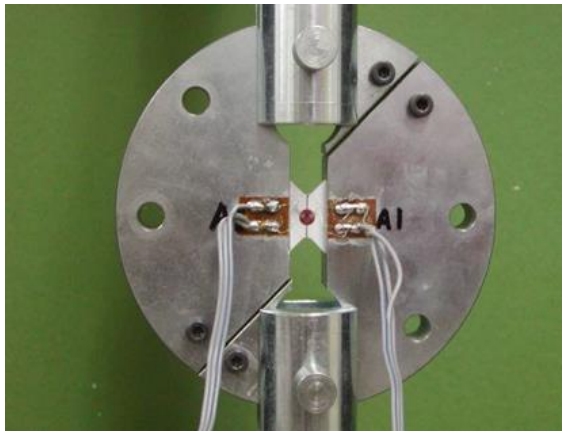


Figure 8 Complete Arcan fixture attachment affixed to holder

2.4 Load Test

The Arcan fixture complete with the butterfly specimen was loaded in tension by using Instron Universal Testing Machine Series IX Model 4206 instrumented with 5 kN load cell. The loading rate was set-up to 1 mm/min and the specimen was loaded to failure. The specimen principal strains (45°) were measured at every 0.1 kN load increment and this was manually recorded by TDS 302 data logger until near

to failure. The applied load and machine displacement were recorded automatically by the computer. In addition a stop watch was used to measure the time to failure for each specimen. As a result, the relationship between the applied shear stress and shear strain could be established by dividing the applied load on the cross-sectional area of the significant section.

3.0 RESULTS AND DISCUSSION

3.1 Shear Properties

It could be observed that the specimen linearly gained shear loads from the start until failure, and exhibited brittle type of failure mode as shown in Figure 9. The fracture mechanism of the epoxy appeared to be initiated from the notch roots of the specimen. This fracture surface were found to be 45° to the loading axis, which was the direction of the tensile principal stress corresponding to the state of the pure shear as shown in Figure 10. Such failure mechanism was also found in the Iosipescu test method on vinylester specimen conducted by Sullivan *et al.* [16], and Arcan test method conducted by Yen *et al.* [17] on Plexiglas specimen.



Figure 9 Brittle failure of ESST-C01 occurred in $\pm 45^\circ$ plane

From the overall results a comparison between shear strength and their respective weight gain and weight loss were established as listed in Table 2.

Table 2 shows that the ESLT-OD sample had the highest shear strength which was 23.85 MPa, followed by ESLT-LB, 21.82 MPa, ESLT-SW, 21.45 MPa; and finally the ESLT-PW, 19.88 MPa. Based on the results, it can be said that the specimens' shear strength was reduced due to the effect of exposure to wet/dry cycles in plain and salt water. The moisture diffusion were calculated from weight measurement before and after the weather exposure.

The chemical chain of the epoxy might be weakened as the water penetrated through diffusion mechanism. Another factor that influenced and affected the shear strength was the oxidation process

for ESLT-OD specimens, as the weight was reduced by about 0.10% by means of yellowish effect on specimens as they were exposed to wet/dry outdoor condition.

Table 2 Overall properties of SelfixCarbofibe adhesive exposure group of specimens

| Exposure condition | Shear strength, τ (MPa) | Shear modulus G (GPa) | Weight gain (%) | Weight loss (%) | Time to failure (s) |
|--------------------|------------------------------|-----------------------|-----------------|-----------------|---------------------|
| ESST-CO | 29.24(9%) | 2.97(8%) | - | - | 66.4 |
| ESLT-LB | 21.82(10%) | 2.59(11%) | 0.11 | - | 50.07 |
| ESLT-OD | 23.85(10%) | 2.70(4%) | - | 0.10 | 45.83 |
| ESLT-PW | 19.88(10%) | 2.64(3%) | 0.25 | - | 48.79 |
| ESLT-SW | 21.45(12%) | 2.54(17%) | 2.67 | - | 47.85 |

Note: The values listed in bracket represent the percentage of deviation from their average fresh specimen.

The epoxy shear strength versus exposure to the test conditions are shown in Figure 10. The shear strength for ESLT-LB, ESLT-OD, ESLT-PW and ESLT-SW were lower than the control sample, ESST-CO. The ESLT-PW sample had the lowest shear strength which was 32% lower than the controlled sample followed by ESLT-SW, 26.6%, ESLT-LB, 25.4%; and finally the ESLT-OD, 18.4%.

The epoxy shear modulus versus exposure conditions is shown in Figure 11. The shear modulus for ESLT-LB, ESLT-OD, ESLT-PW and ESLT-SW were also lower than the controlled sample, ESST-CO. The ESLT-SW sample had the lowest shear modulus, which was 14.5% lower than the control sample, followed by ESLT-LB (12.8%), ESLT-PW (25.4%) and finally the ESLT-OD (9.0%).

In this study, the material is assumed as homogeneous and isotropic. Unfortunately, this condition did not exist in SelfixCarbofibe adhesive epoxy due to the formation of micro-porosity (Figure 12). The testing method and procedure also played important roles in obtaining accurate test results. For

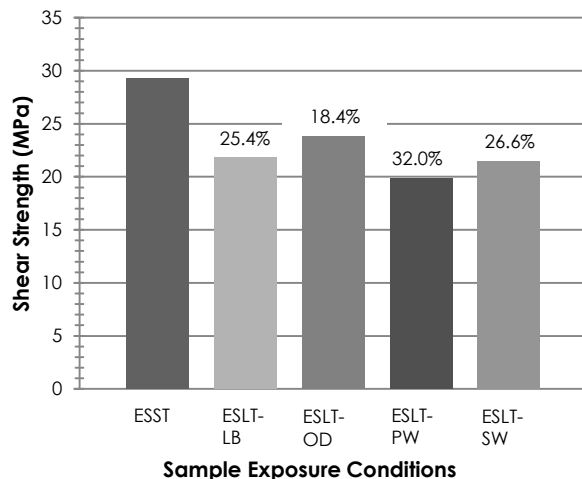


Figure 10 Shear strength values and reductions after exposure to tests conditions

example, the bonding of the specimen to the Arcan fixture and the test rig alignment also had a significant effect on the experimental results [17]. Each manufacturer has its own testing specification which may differ from one to the other. This is very much dependent on the manufacturer's definition and procedure of testing. More importantly, their testing method refers directly to the materials actual application.

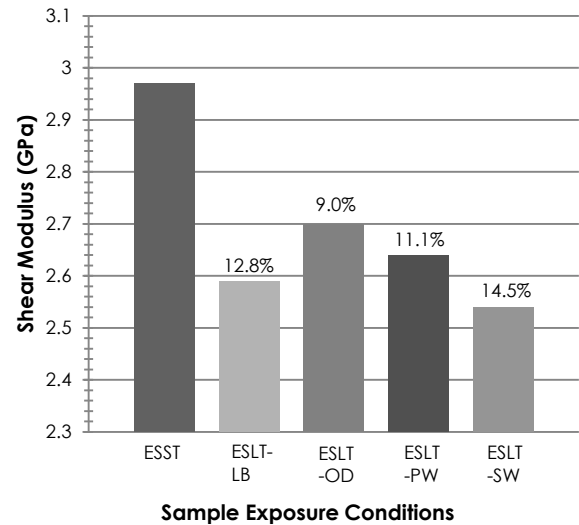


Figure 11 Shear Modulus values and reductions after exposure to test conditions

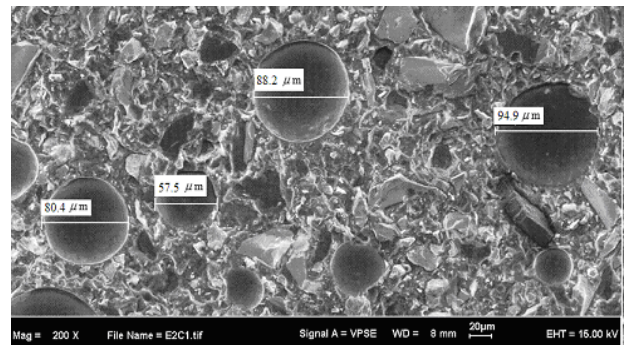


Figure 12 The formation of micro-porosities size in ESST-CO.

3.2 Chemical Elements in SelfixCarbofibe Adhesive

After the samples had been exposed to the designated conditions, selected specimens underwent scanning process by using the FESEM microscope. The main objective was to determine the changes of base elements in each sample. The base elements content for each sample are presented in Table 3.

This microstructure analysis is also an important subject to focus on in order to determine the factors that may affect the strength of epoxy adhesive due to tropical exposure conditions. An investigation was made to determine the influence of environment conditions (tropical conditions) on the epoxy micro

structural system. For that purpose, the fracture parts and specimen surfaces were analysed using Field-emission Scanning Electron Microscope (FESEM).

From Table 3, silicon was the main filler in the epoxy system which was added to enhance the bond properties. However, the composition of silicon in each specimen was distributed unevenly due to their geometrical non-uniformity. Element contents such as oxide, chlorine and silicone in specimens did not differ much due to the experimental exposures.

Table 3 Elements weight percentage for SelfixCarbofibe adhesive after exposure

| Specimen | Element Weight (%) | | | | | |
|-----------|--------------------|-------|-------|------|------|------|
| | O | C | Si | Ti | Mg | Cl |
| ESST-C02 | 35.66 | 49.30 | 12.35 | 1.46 | 0.29 | 0.95 |
| ESLT-LB01 | 34.35 | 47.97 | 14.54 | 2.22 | 0.19 | 0.73 |
| ESLT-OD03 | 35.01 | 50.94 | 11.30 | 1.75 | 0.30 | 0.70 |
| ESLT-PW05 | 34.35 | 52.14 | 11.02 | 1.84 | 0.08 | 0.57 |
| ESLT-SW03 | 35.83 | 49.97 | 11.76 | 1.52 | 0.22 | 0.71 |

The microstructure analysis results using FESEM for all selected exposure specimens are shown in Figures 13 to 17. Figure 13 shows the standard texture of the epoxy's top surface that experienced no exposure effects (i.e. control specimen). It shows perfect bonding between epoxy matrix and silicon based filler that was used to enhance the mechanical properties of the epoxy system. Figure 14, clear shows that the ESLT-LB material texture experienced environmental effects due to the surrounding air that contained high moisture level in the laboratory. The significant effect of the six months exposure period on the ESLT-OD specimen was the formation of crack line that was probably caused by oxidation process as shown in Figure 15. The specimen also experienced unpredictable chemical effects due to outdoor weathering or climate changes such as temperature fluctuations, presence of high humidity level, reaction to mineral rain content, ultraviolet radiation and others. The metal elements composed in the epoxy system as shown in Table 2 also have the tendency to react with oxygen (O_2) in the air and formed metal oxides such as MgO and TiO_3 (in the form of dust) [18]. These effects resulted in reduced weights of the specimens.

The effect of plain water on ESLT-PW top surface can be seen in Figure 16. There was a sign of erosion and cracking on the material surface. Generally, epoxy adhesives are prone to water absorption because they possess polar sites that attract water molecules through diffusion mechanism. Water molecules are typically hydrogen bonded to hydrophilic groups of cured epoxy mainly hydroxyl and amine groups [19–21]. The absorption of water has greatly influenced the

strength of the epoxy structure. Water might have penetrated the epoxy system by diffusion or capillary action through cracks or porosity. The water molecules entered into the epoxy material bond with the polymer through hydrogen bonding. By this mechanism, the water was able to disrupt the inter chain Van der Waals forces inside the network producing an increase in segmental mobility [22, 23]. This hydrolysis of the bisphenol-A epoxy by water absorption is a form of chemical degradation of the matrix.

The effect of saltwater exposure condition for ESLT-SW specimen can be seen in Figure 17, where the formation of salt ($NaCl$) that adhered to the specimen surface could be observed. This might be caused by a volume of saltwater that has already saturated in the specimen due to absorption process. The specimen has experienced the highest weight gain which was about 2.67% as shown in Table 2. The density of saltwater is higher than plain water and has caused more absorption into the specimen. The chemical reaction formed between $NaCl$ ions and epoxy polymer system indicate the possibility that the shear property of the specimen has been reduced. The epoxy that initially consisted of siklo bonds was reduced to single bond due to the chemical reaction between Na^+ Cl^- and epoxy. It is known that the strength of a single bond is actually weaker than siklo bond.

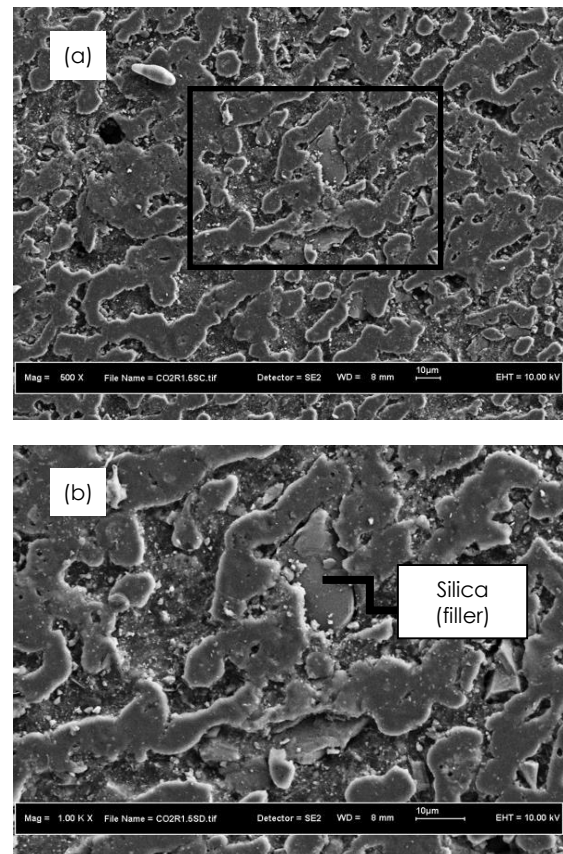


Figure 13 ESST-CO surface texture at (a) x500 magnification and (b) x1000 magnification

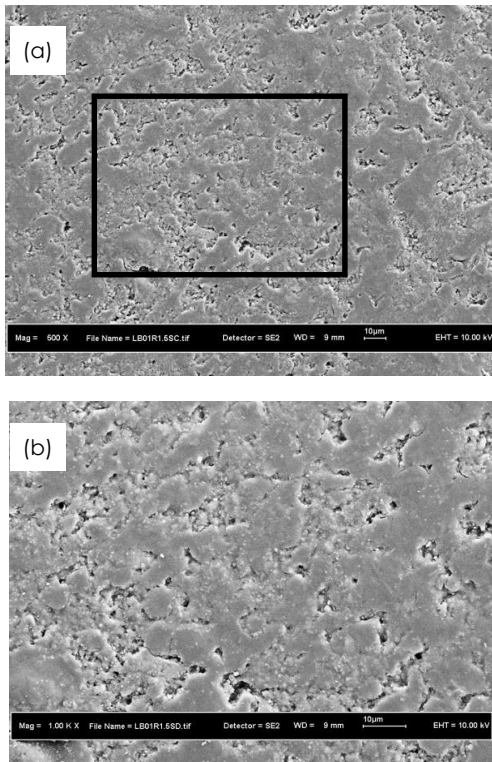


Figure 14 ESLT-LB surface texture exposed to laboratory condition at (a) x500 magnification and (b) x1000 magnification.

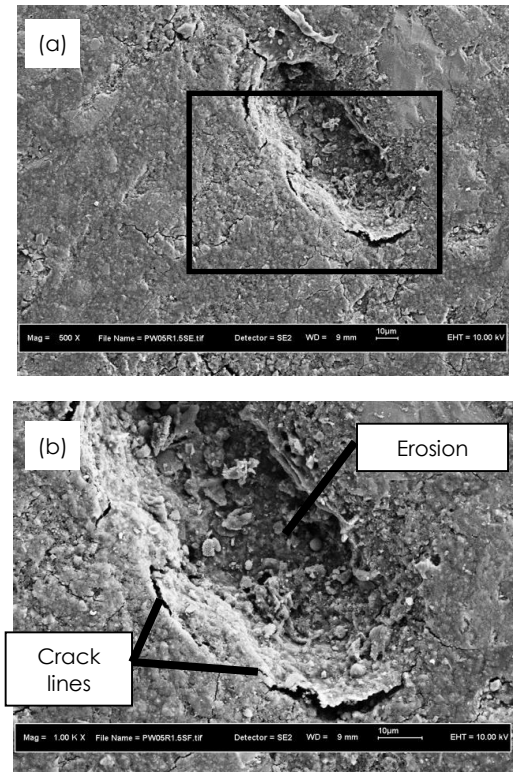


Figure 16 ESLT-PW surface texture exposed to plain water (wet/dry) condition at (a) x500 magnification and (b) x1000 magnification.

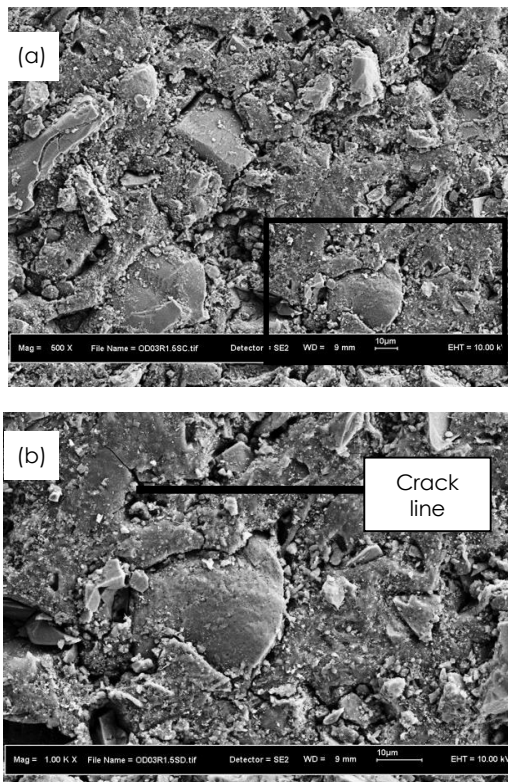


Figure 15 ESLT-OD surface texture exposed to outdoor condition at (a) x500 magnification and (b) x1000 magnification.

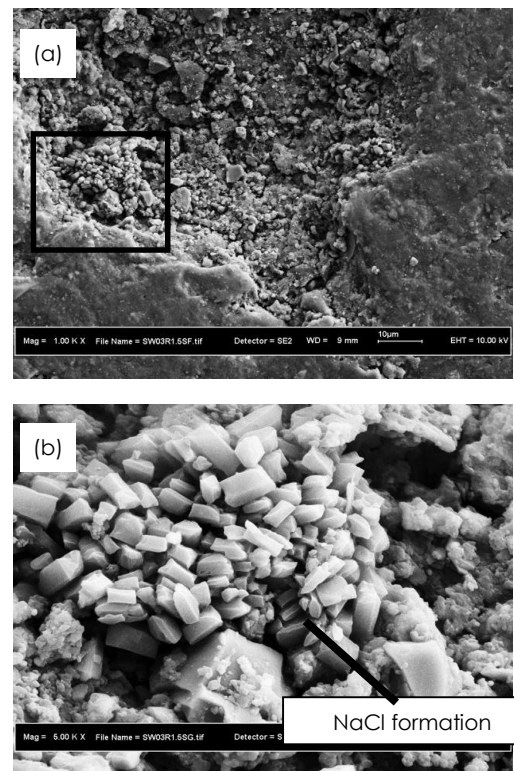


Figure 17 ESLT-SW surface texture exposed to salt water (wet/dry) condition (a) x1000 magnification and (b) x5000 magnification.

4.0 CONCLUSIONS

The major conclusions that can be made from this study are summarised as follows:

- i. A significant section of the butterfly specimen had proven that the Arcan test method was reliable as the shear stress and strain relationship was linearly propagated. The results also show that the strains in both directions were not perfectly symmetrical for some of the specimens possibly resulting from micro-porosity effects. The overall results can be accepted as the average strain difference was only about 7%.
- ii. The formation of micro-porosity during casting process was difficult to eliminate due to the geometrical effect of epoxy composition materials that led to air entrapment which finally reduced the epoxy cross-sectional area of the significant section. The effect was also a factor that accelerated the moisture diffusion process into the epoxy system.
- iii. All specimens failed in brittle form. The load test results show that the exposure conditions had influenced the shear strength and modulus of the epoxy system especially for those exposed to plain and salt water conditions. The saltwater exposure produced the most significant weakening (17% reduced) of the shear strength of the material.

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