- Technical Paper -

MOISTURE EFFECT ON THE RESIN AND INTERFACIAL FRP-CONCRETE BOND PROPERTIES

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ABSTRACT

Long term durability of FRP-concrete bond and its constituent materials under the influence of continuous moisture was investigated by an experimental study. The results obtained from both material and bond test revealed excellent water resistant performance even after a year of continuous exposure in water. The only signs of deterioration due to water were observed at the failed surfaces. The failure shifted more towards primer-concrete interface from concrete cohesion failure with increase after immersion.

Keywords: FRP, bond, moisture, interface, concrete

1. INTRODUCTION

Even though the application of Fiber Reinforced Polymer (FRP) composites in aerospace, automotive and marine construction have been started a long time ago, the use in civil construction was started few decades ago. The popularity of these FRP materials in construction industry is mainly due to its several advantages such as high strength and stiffness, light weight, easiness to application, and excellent corrosion resistance etc. over previously used other traditional materials. With the wider applications, the long term durability issues of the materials and the whole composite system needs to be investigated. One of the potential durability related issues in marine environment was identified as the moisture by many past researchers [1-5] .

It has been reported that the moisture could affect the adhesive bond in three ways which are 1) degradation of the bulk properties chemically, physically and mechanically, 2) degradation of the adherent/adhesive interfacial adhesion properties and 3) degradation of the properties and induce dimensional changes of adherents [6]. It is also very important to understand that the selection of FRP and resin materials, ageing conditions and testing methods also vastly affects the bond durability due to which different conclusions could be found in available literatures.

Therefore, the current study focuses on improving the understanding on the long term effect of water on the epoxy resins and the bond at FRP-concrete interfaces.

2. TEST PROGRAMS

2.1 Materials

(1) Primers and impregnating resin

Three room temperature epoxy resins were used in this study which are named as Epoxy-E, Epoxy-F and Epoxy-R. Epoxy-E is a two component epoxy primer with resin and hardener part mixed in the ratio of 4:1 by weight. The resin part is formulation of Bisphenol-A and Bisphenol-F type epoxy resin whereas hardener is combination of polythiol and polyamines. This is a high modulus concrete bonding primer usually used in application of steel rod anchorage to concrete, old-new concrete bonding, wet surface applications etc. Epoxy-F and Epoxy-R are also two component adhesives with resin and hardener part mixed in the ratio of 2:1 by weight. The resin part is formulation of Bisphenol-A type epoxy resin whereas hardener part consists of blends of polyamines. Epoxy-F is an epoxy primer whereas Epoxy-R is the impregnating resin for the FRP sheet. Epoxy-R has an additional silica component than Epoxy-F.

(2) Continuous fiber sheet (CFS)

Unidirectional CFS made of carbon fiber is used with the design thickness of 0.111 mm and Fiber Areal Weight (FAW) of 200 g/m^2. The tensile strength and modulus are 3400 N/mm^2 and 2.45×10^5 N/mm^2 respectively.

(3) Concrete

The early strength concrete of mean cylindrical compressive strength 39.4 MPa was used in this study. The mean was taken from samples tested at different ages (0, 1, 3, 4, 6 months) after the casting.

2.2 Preparation of Specimens

The epoxy tensile specimens were prepared by mixing resin and hardener in the specified ratio which

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was then put in the silicon mold to form a dog bone shaped specimen as shown in Fig. 1. All three epoxy specimens were prepared in similar manner in accordance with JIS K 7113 (1995).

In order to measure apparent shear strength of the resins, lap shear specimens were prepared by bonding two pretreated steel plates with the small quantity of epoxy resin as shown in the Fig. 2. The specimens were prepared with Epoxy-E, Epoxy-F and Epoxy-R following JIS K 6850 (1999).

For FRP-concrete bond specimens, the concrete blocks were casted and cured for 28 days under moist condition in the laboratory. The bonding surface was grinded by disk sander to remove the thin mortar layer until aggregates became visible. The amount of surface preparation was visually judged and attempt was made to make uniform surface preparation throughout the specimens. After necessary surface preparations, the surface was cleaned by compressed air to remove small dust particles from the surface. Then a thin uniform layer of primer was applied on the surface with a roller and allowed to cure for 24 hours. Finally, the CFS was impregnated with the resin and bonded to the concrete layer to form a composite. The details of the specimen are shown in Fig. 3. The effective bond length of 200mm was adopted to ensure that it exceeds the effective bond length which is found to vary from 50mm to 100mm for the given stiffness value [7]. All the three kinds of specimens were cured for a month before exposing them into environmental exposures. The study parameters are summarized in Table 1.

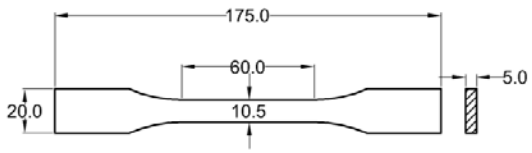


Fig. 1 Details of tensile resin specimen (unit: mm)

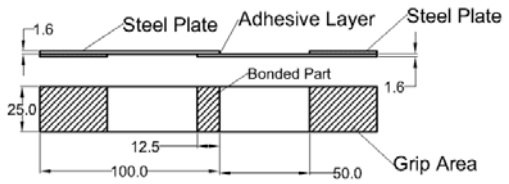


Fig. 2 Details of shear specimen (unit: mm)

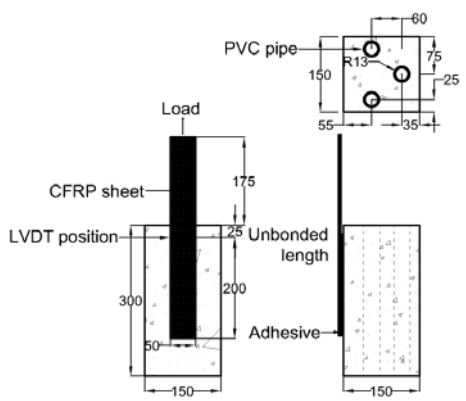


Fig. 3 Details of bond specimen (unit: mm)

Table 1 Study parameters

Specimen	Resin	Conditions	Exposure duration (Months)
Tensile/ Shear	Epoxy-E/ Epoxy-F/ Epoxy-R	Room	0,1,2,3,4,6
		Water immersion	1,2,3,4,6,9,12
Bond (Type-E)	Epoxy-E + Epoxy-R	Room	0,1,2,3,4,6
		Water immersion	1,2,3,4,6,9,12
Bond (Type-F)	Epoxy-F + Epoxy-R	Room	0,1,2,3,4,6
		Water immersion	1,2,3,4,6,9,12

2.3 Exposure Conditions

Usually, the study of environment related deterioration phenomenon takes very long period of time. Due to the time limitation, the deterioration process is accelerated in the laboratory testing by continuous immersion of specimens into water maintained at a constant temperature of 20 °C. The arrangement of the concrete water pool is shown in Fig. 4. Tap water is continuously circulated in the pool with a submersible pump in order to maintain uniform water temperature throughout the pool. After the required ageing period, specimens were taken out from the water and tested immediately with wet conditions.

The remaining set of specimens was put in ambient conditions inside the laboratory. These sets of specimens are considered as the control specimens for the study.

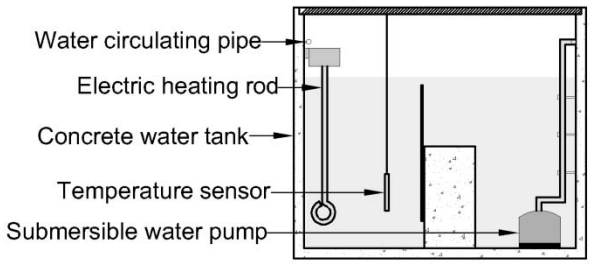


Fig. 4 Schematic of exposure tank

2.4 Testing Procedure

As shown in Fig. 5, the specimen was mounted on the universal testing machine and reaction steel plate was put on top of it. Three long bolts were inserted through the preset plastic pipes inside the concrete specimens which were fixed at the base of the machine. The specimen was adjusted in position to make sure that the FRP-concrete bond line is aligned with the center line of the upper loading grip. Strain gauges were attached on some of the specimens to record the strain behavior of the FRP sheet and Linear Variable Displacement Transducer (LVDT) was positioned at the starting point of the bonding zone to measure the loaded end slip between the concrete and FRP sheet. The loading speed of the upper grip was set as 0.2 mm/min.

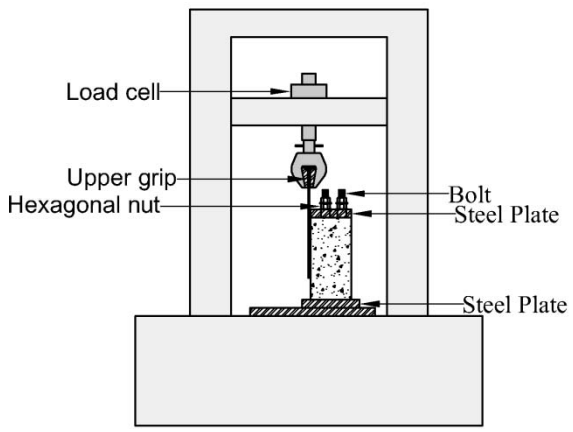


Fig. 5 Test arrangements for the bond specimen

3. RESULTS AND DISCUSSION

3.1 Moisture effect on mechanical properties of resin

The moisture absorption characteristics of resins are very important. There are many reported harmful effects of water on the properties of the epoxy resins such as plasticization, hydrolysis, cracking and crazing etc [8]. Usually, the amount of water absorbed by the resin material is taken as an indicator to quantify the damage in the mechanical properties of the resins. To understand the behavior of water diffusion into the resins, the moisture absorbed by tensile resin specimens was recorded at different immersion durations.

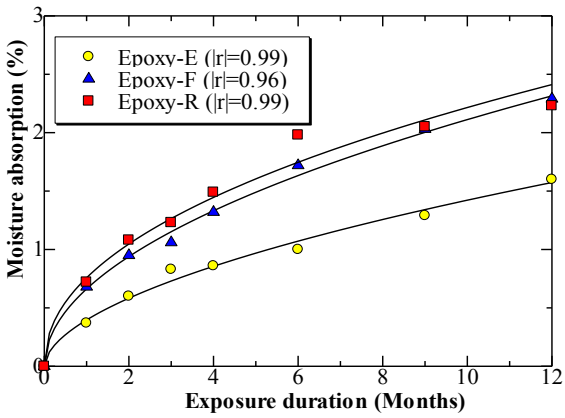


Fig. 6 Moisture absorption by the tensile resin specimens

As seen in Fig. 6, initially the moisture diffusion, into the resin specimens were higher due to quickly absorption of water through all the surfaces, but as the specimens started getting partially saturated, the diffusion rate decreased to a constant rate until now. But even after 12 months of continuous immersion in water, the result clearly indicates that the equilibrium state has not reached yet.

The moisture absorbed by the specimens after 12 months of water immersion was 1.6%, 2.3% and 2.2% for Epoxy-E, Epoxy-F and Epoxy-R respectively. Au [1] also recorded similar range (1.14% and 2.15%) of moisture uptake by epoxy resin specimens when amine hardener epoxy resins were used. In contrast, Sciolti *et al.* [9] reported the absorption range from 4.7% to 8.2% after 24 weeks of immersion at 23±2 °C but the information on the type of epoxy resin was not mentioned.

The moisture absorption behavior shown by Epoxy-F and Epoxy-R are very close probably due to their similar chemical components and compositions. Whereas, the water absorption by Epoxy-E is comparatively lower than the other two resin specimen types which could be due to existence of polythiol components in the curing agent which has greater water resistance properties.

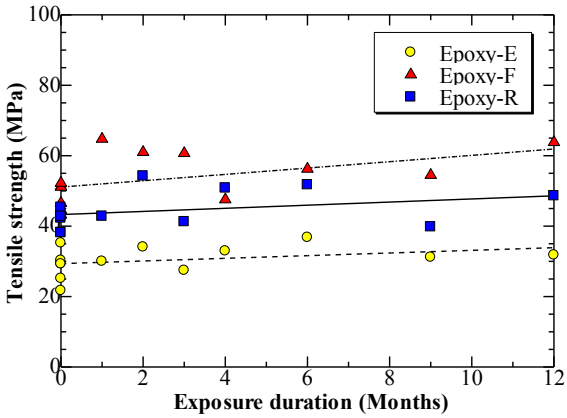


Fig. 7 Relationship between tensile strength and the exposure duration

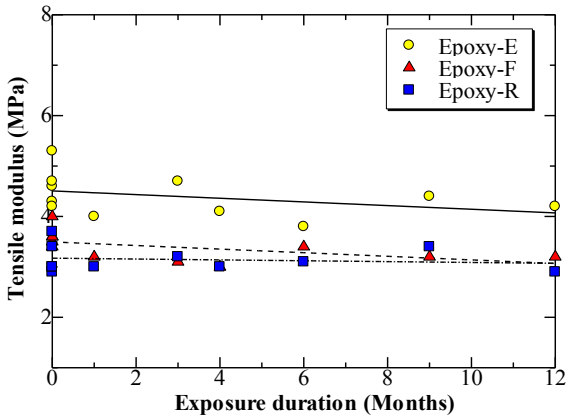


Fig. 8 Relationship between tensile modulus and the exposure duration

In this study, the investigation of moisture effect on the material properties is carried out by comparing the mechanical properties of the materials subjected to different lengths of exposure duration. Unlike other published results which showed significant reduction of strength and stiffness of resins due to continuous exposure in water [1, 9], the obtained result show different nature with excellent resistance to water in the given exposure conditions. Fig. 7 and Fig. 8 show the relationship of the tensile strength and stiffness for different epoxy resins with the exposure durations. For all three cases, even until 12 months of exposure in water, no signs of reduction on the tensile properties of the resins were observed. The reason for such excellent resistance properties could either be due to good water resisting properties of the adhesives or could be the

absorbed moisture may not be sufficient yet to cause any damages. Brewis *et al.* [10] and Kinloch [11] presented the concept of critical moisture content according to which there exists a critical water concentrations within the adhesive, below which water-induced damage of the joint will not occur. The adhesive chosen by Brewis was DGEBA cured with 1,3-Diaminobenzene. According to their studies, critical water content was found out to be 1.35% to 1.45%. But this critical moisture may vary based on the types of epoxy resins. So, this critical value of moisture may not have reached in the resin used for this study. The results obtained from the tensile test well support those from the shear test of the resins. As presented in Fig. 9 the effect of water on the shear strengths of the epoxies were found insignificant. For deeper understanding, chemical compositions of the resins need to be determined and understanding on how such components would interact with water needs to be determined.

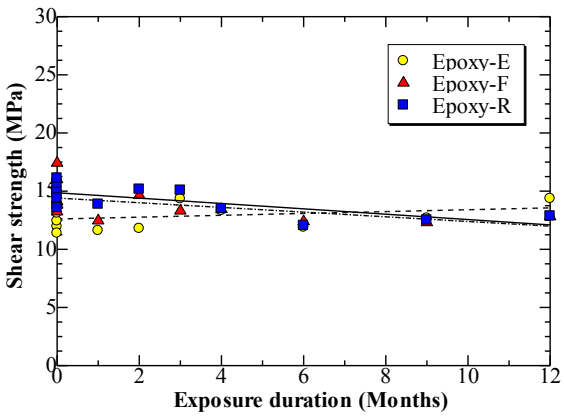


Fig. 9 Relationship between shear strength and the exposure duration

The commercial room temperature cured epoxy resins usually consist of two components namely resin part and hardener part which is the curing agent. The resin part is mainly composition of Diglycidyl Ether of Bisphenol-A (DGEBA) which is formed by reaction between Bisphenol A and epichlorohydrin but the hardeners are blends of several different components such as polyamines, polyamides, polysulphide etc. This blending of different chemicals and addition of various modifiers such as flexiblizers, tougheners, diluents, fillers, thixotropic agents etc. makes each commercial epoxy with the unique characteristics. These modifications on the resin properties are done to improve gel time, increase curing rates, achieve better heat resistance and greater resistance to chemicals and water absorption. The properties of the resin could be adjusted based on the user-intended application as a result of which durability related studies conducted by using resins from different manufacturers may yield vastly different conclusions.

3.2 Moisture effect on concrete strength

Fig. 10 gives the relation between concrete compressive strength and immersion duration. As the specimens were tested immediately after removal from the water tank, the reduction in concrete strength could be due to intensified pore water pressure resulting into micro cracks generated during application of external forces [12].

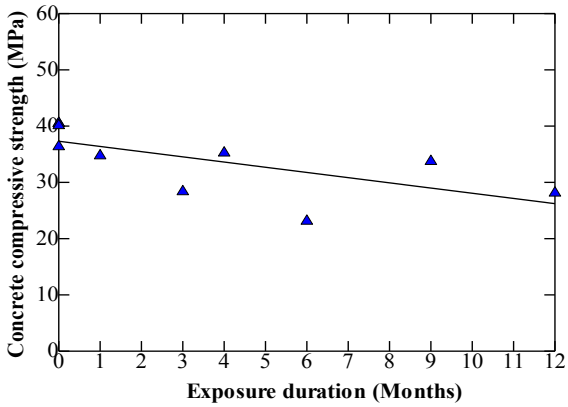


Fig. 10 Relationship between concrete compressive strength and exposure duration

3.3 Moisture effect on FRP-concrete bond

Fig. 11 shows the relationship between ultimate bond strength and the exposure duration for Type-E and Type-F specimens. Even though some experimental scatter exists in the results, no obvious reduction in the bond strength was observed up to the current maximum immersion duration indicating no serious harmful effects of water on the FRP-concrete bond. The excellent mechanical peformance shown by the resins supports the above bond behavior. However it is to be noted that the failure modes changed after water immersion for both specimen types.

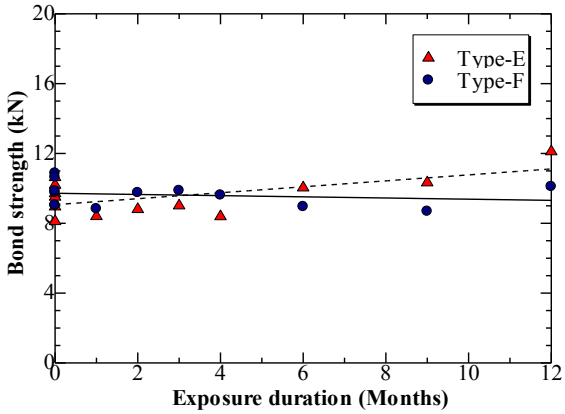


Fig. 11 Relationship between bond strength and the exposure duration

3.4 Analysis of failure surfaces

The observation of failed surfaces gives good idea on the propagation of the crack path. The comparison of failed FRP sheets at various durations of exposure is shown in Fig. 12 and Fig. 13 respectively. Careful observation indicates difference in failure after exposure in water. Usually the shear strength of the structural adhesives are very high compared to that of concrete [13] so failure mostly occurs at the concrete

side provided that proper surface preparation is done. The failure before the immersion was mostly at the thin concrete layer which shifted to primer-concrete interface after immersion as demonstrated in Fig. 14. Such shift in failure modes after interaction with water were also observed by other researchers [3-5]. This phenomenon is observed in both specimen types indicating some effects of water on the bond at primer-concrete interface. However, differences exist between two cases; Epoxy-E is specifically designed for concrete to concrete bonding so adhesion with the concrete is stronger for Epoxy-E than Epoxy-F which is evident from the failed FRP sheets as shown in Fig. 12 (0 month). With the increase in duration of immersion, the water molecules destroyed the adhesion bond between primer and concrete which caused the change in failure mode. But as the bonding force between concrete and primer is stronger in case of Type-E specimens, the shifting of failure mode to adhesion took longer time than the Type-F specimens.

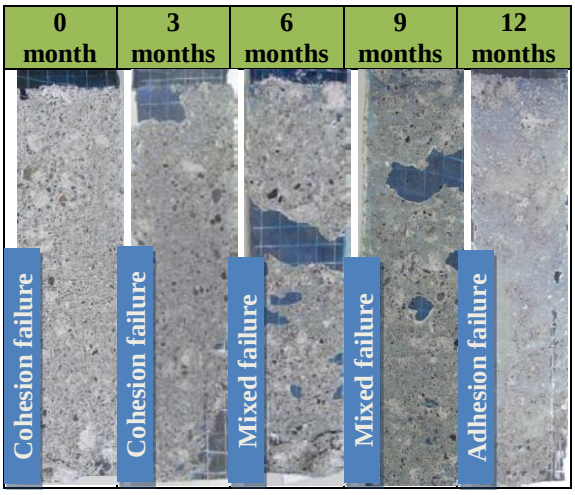


Fig. 12 Comparison of failure surfaces after at different exposure durations (Type-E)

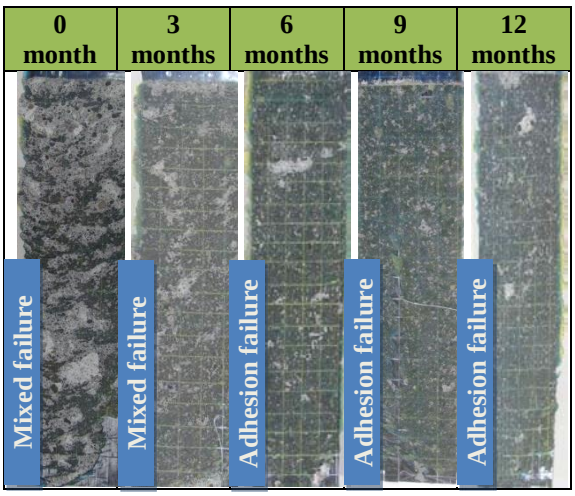
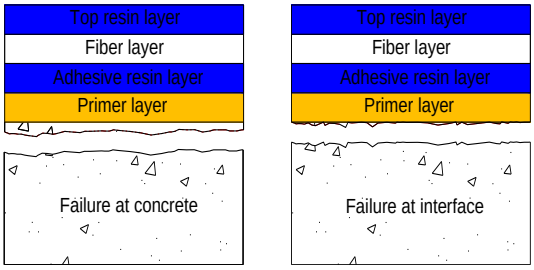


Fig. 13 Comparison of failure surfaces after at different exposure durations (Type-F)



Before exposure After exposure

Fig. 14 Demonstration of failure modes

The shift of failure to primer-concrete adhesion from concrete cohesion could have two different possibilities. The first could be due to increase in shear strength of the substrate concrete as a result of post curing of the concrete thus making the adhesion layer more vulnerable to failure. But if we consider the results obtained from the concrete cylinder test as reference (Fig. 10), then it clearly indicates that the strength is not further increased despite continuous immersion in water for several months which denies the above hypothesis. This leads to conclusion that the adhesion bond becomes weaker due to interaction of water molecules with the epoxy-concrete bond. To understand more precisely, the bond mechanism at the interface needs to be clarified. In the epoxy-concrete bonding, the primary bonds at the interface are mechanical interlocking and intermolecular forces mainly hydrogen bonding. When this bonding system is introduced to wet environments, the moisture could diffuse through various different ways such as through concrete pores, adhesives, cracks or defects etc. and reach the interface region. This moisture at the interface could interact with specific hydrophyllic functional groups, such as hydroxyl or amine in epoxy resin and disrupt the hydrogen bond [14]. The evidence of such breakage of hydrogen bond due to interaction with water were observed by some researchers [15, 16]. Similarly, in the present case, the water molecules at the interface might have combined with some of the epoxies destroying the hydrogen bond. The visualization of the deterioration process is explained schematically in Fig. 15. Before the exposure, the primer and concrete molecules are well bonded by adhesion forces, which after exposure in water, are partially destroyed at some locations. Despite breakage of bond at few locations, it is surprising to see almost unaffected bond strength during the entire study. The possible reason is tried to explain in the following manner. At the beginning, the cohesion strength is lesser than the adhesion strength which caused the failure to always occur at the thin concrete layer but with the introduction of water, the cohesion strength remained unaffected but the adhesion strength decreased due to breakage of the bond by water molecules (as demonstrated in Fig. 15) until a point where it becomes just lower than the cohesion strength. At this point, primer-concrete interface becomes the weak region so as a result, failure occurs at the interface thus changing the failure mode but due to very similar

cohesion and adhesion strengths, the bond strengths were almost unchanged even after the exposure. However, these hypothesis need to be further investigated in more detail in future.

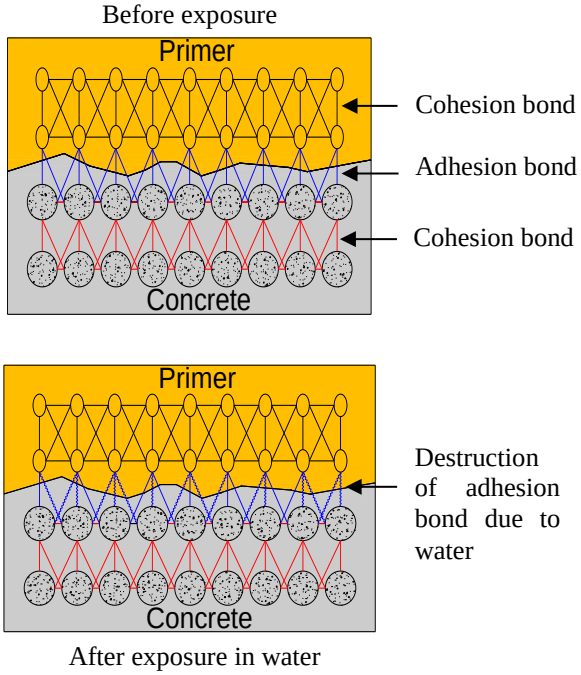


Fig. 15 Visualization of bond deterioration after water immersion

4. CONCLUSIONS

FRP-concrete bond and its key constituent materials were investigated under the influence of water by an experimental study and following important conclusions could be drawn out.

1) The results clearly demonstrated that the materials chosen for the study showed excellent performance despite continuous exposure to water for a year.
2) A concrete epoxy primer was also found suitable for FRP application. The primers with polyamine and polythiol components seem to show good resistance to water.
3) The relationship between bond strength and the exposure duration was obtained. Despite change in failure modes, the effect on the bond strength is minimal.
4) Some signs of water related deterioration was observed at the primer-concrete interface. The failure mode shifted from concrete cohesion failure to primer-concrete adhesion failure after water immersion.

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REFERENCES

[1] Au C. Moisture degradation in FRP bonded concrete systems: an interface fracture approach Doctoral dissertation, Massachusetts Institute of Technology; 2005.
[2] Lau D, Büyüköztürk O. Fracture characterization of concrete/epoxy interface affected by moisture. Mechanics of Materials. 2010;42(12):1031-42.
[3] Tuakta C, Büyüköztürk O. Deterioration of FRP/concrete bond system under variable moisture conditions quantified by fracture mechanics. Composites Part B: Engineering. 2011;42(2):145-54.
[4] Benzarti K, Chataigner S, Quiertant M, Marty C, Aubagnac C. Accelerated ageing behaviour of the adhesive bond between concrete specimens and CFRP overlays. Construction and Building Materials. 2011;25(2):523-38.
[5] Dai J, Yokota H, Iwanami M, Kato E. Experimental Investigation of the Influence of Moisture on the Bond Behavior of FRP to Concrete Interfaces. Journal of Composites for Construction. 2010;14(6):834-44.
[6] Petrie EM. Epoxy adhesive formulations: McGRAW-HILL; 2006.
[7] Dai J. Interfacial Models for Fiber Reinforced Polymer (FRP) Sheets Externally Bonded to Concrete Doctoral dissertation, Hokkaido University; 2003.
[8] Mays GC, Hutchinson AR. Adhesives in civil engineering: Cambridge University Press; 1992.
[9] Sciolti M, Frigione M, Aiello M. Wet Lay-Up Manufactured FRPs for Concrete and Masonry Repair: Influence of Water on the Properties of Composites and on Their Epoxy Components. Journal of Composites for Construction. 2010;14(6):823-33.
[10] Brewis DM, Comyn J, Raval AK, Kinloch AJ. The effect of humidity on the durability of aluminium-epoxide joints. International Journal of Adhesion and Adhesives. 1990;10(4):247-53.
[11] Kinloch AJ. Interfacial Fracture Mechanical Aspects of Adhesive Bonded Joints—A Review. The Journal of Adhesion. 1979;10(3):193-219.
[12] Bazant ZP, Prat PC. Effect of Temperature and Humidity on Fracture Energy of Concrete. ACI Materials Journal. 1988;85(4):262-71.
[13] Tu L, Kruger D. Engineering properties of epoxy resins used as concrete adhesives. ACI Materials Journal. 1996;93(1).
[14] Bellenger V, Verdu J, Morel E. Structure-properties relationships for densely cross-linked epoxide-amine systems based on epoxide or amine mixtures. Journal of materials science. 1989;24(1):63-8.
[15] Zhou J, Lucas JP. Hygrothermal effects of epoxy resin. Part I: the nature of water in epoxy. Polymer. 1999;40(20):5505-12.
[16] Lefebvre D, Takahashi K, Muller A, Raju V. Degradation of epoxy coatings in humid environments: the critical relative humidity for adhesion loss. Journal of adhesion science and technology. 1991;5(3):201-27.