



Title	Durability of FRP Concrete Bonds and Its Constituent Properties under the Influence of Moisture Conditions
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Citation	Journal of materials in civil engineering, 27(2), A4014009 https://doi.org/10.1061/(ASCE)MT.1943-5533.0001093
Issue Date	2015-02
Doc URL	http://hdl.handle.net/2115/58490
Type	article (author version)
File Information	[JS 140206] ASCE_Manuscript_Justin_Revision.pdf



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1 Submission to the Special Issue of PLSE 2012 on Journal of Material for Civil Engineering

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3 Durability of FRP-concrete bond and its constituent properties under the influence of
4 moisture conditions

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24 **Abstract:**

25 Strengthening by Fiber Reinforced Polymers (FRPs) is one of the most common solutions to
26 the ageing infrastructures. However, long term durability of such systems under different
27 environment conditions need to be understood properly before widely adopting these
28 methods in the field. This paper presents research outcomes from an experimental program
29 carried out to determine the influence of moisture on the durability of the bond between FRP
30 and concrete along with its constituent materials. Performance was evaluated through single
31 lap shear bond test and various kind of test on the resin samples including water absorption,
32 mechanical characterization and glass transition temperature (T_g) analysis. The specimens
33 were exposed to continuous water immersion and wet-dry cycles for a maximum period up to
34 24 and 18 months respectively.

35 The results show some deterioration on the material and the bond properties in both exposure
36 conditions. The bond strength decreased up to 32 and 12% for high and normal strength
37 concrete substrate respectively after continuous immersion indicating key role of substrate in
38 the durability of bond. As a result of exposure, failure propagated towards the primer-
39 concrete interface region. The failure mode changed from concrete cohesion to primer-
40 concrete mixed failure for normal strength concrete and from mixed failure to complete
41 adhesion failure in case of high strength concrete. These shifts in failure patterns are mainly
42 due to destruction of adhesion bond caused by water molecules at the interface. However, it
43 is found that the loss of adhesion bond is somehow compensated by the good mechanical
44 interlocking obtained by proper surface roughness condition of the substrate. Finally, the
45 effects of water were also investigated in interfacial bond stress-slip laws and fracture
46 energies.

48 **Keywords:** fiber reinforced polymer (FRP); concrete; bond; moisture; interface; continuous
49 immersion; wet-dry cycles

50 INTRODUCTION

51 Externally bonded carbon fiber reinforced polymer (FRP) composites are extensively being
52 used in rehabilitation and strengthening of civil infrastructures such as bridge decks, girders,
53 buildings, marine structures etc. The main advantages of this composite include high strength
54 and stiffness, light weight, ease of installation, higher durability against corrosion etc.

55 Although FRP composites are considered as highly durable, some of the recent studies have
56 reported harmful effects when subjected to long-term environmental exposures such as
57 moisture (Au 2005; Au and Büyüköztürk 2006), temperature (Leone et al. 2009), chemical
58 solutions (Choi et al. 2012), freeze-thaw cycles (Subramaniam et al. 2008), UV (Dolan et al.
59 2010) etc. In marine environment, moisture is considered as one of the most serious
60 environmental factors which can deteriorate the integrity of bond between FRP and substrate
61 concrete along with its constituent materials. Consequently, durability of composite system is
62 governed by two major factors which are behavior of individual material and bond between
63 these different materials in the system.

64 In a wet layup system, primary components of FRP-concrete composite are resins, fiber sheet
65 and concrete substrate. The carbon fiber sheets is considered as highly durable material with
66 a very good resistance against harsh environments. Several authors have already reported
67 insignificant effect of moisture and other corrosive solutions on carbon fiber composites
68 (Saadatmanesh et al. 2010; Sciolti et al. 2010). This shifts our attention to the substrate
69 concrete and the resins. In case of concrete, it is generally observed that the mechanical
70 properties degrade with degree of saturation (Bazant and Prat 1988). Reduction in
71 compressive strength was found to be as 30% to 40% (Konvalinka 2002) when cured for 720
72 days. Therefore, understanding on the mechanical behavior of concrete under moisture
73 conditioning is very important. Another most crucial component of the composite system is

74 the resin. Thermosetting epoxy resin is the most frequently used adhesive under normal
75 application. Generally, there are 3 different distinct layers of resin in a composite namely
76 primer, saturant/impregnating matrix and putty. These layers are chosen for the composite
77 system based on requirement of the FRP system and manufacturer recommendation. The
78 reported harmful effects of water on the properties of these epoxy resins are plasticization,
79 hydrolysis, cracking and crazing that can directly affect the mechanical properties (Mays and
80 Hutchinson 1992). These effects are often found to be linked with the absorption of water by
81 the resins. Plasticization and hydrolysis of the resins are often caused due to decrease in T_g of
82 the resin whereas cracking and crazing are linked to the swelling of epoxy resins caused by
83 moisture absorption. Wright (1981) found out that above 1% moisture content, there exists a
84 linear relationship between amount of water absorbed and overall volume or thickness change.
85 Table 1 summarizes the results of some previous literatures showing significant effect of
86 water and other exposure conditions on the mechanical and thermal properties of different
87 epoxy resins. It is clear that there are some harmful effects of water on the resin properties.
88 However, the degree of damage caused by such exposures show a big variation among the
89 researchers. Such differences are largely induced by the use of different materials (mainly the
90 epoxy resins) and testing techniques. But in overall, durability of the composite system is not
91 only governed by these resins but also the interaction between different layers.

92 One of the most important layers in a composite system is the resin-concrete interface, as
93 most of the previous studies have observed failures occur around this region. There are many
94 factors which may affect this interface such as nature of the substrate material, the method of
95 surface preparation, primer and its interaction with substrate, viscosity of the resin, type of
96 FRP system, bond at the interface etc. These all factors make the interface region very
97 complex and difficult to understand the mechanism and durability of the system. Table 2
98 gives summary of some of the studies conducted on the durability of FRP-concrete bond at

99 different exposure conditions and durations. As it can be seen that most of the studies have
100 shown large reduction in bond properties and change in failure mode after such exposures.
101 Apart from these, the moisture diffusion information is equally important in FRP
102 strengthened concrete systems. Some of the researchers have tried to find moisture content at
103 the interface and its correlation with the deterioration due to moisture (Au and Büyüköztürk
104 2006; Ouyang and Wan 2008). Ouyang et al. (2008) have derived the moisture diffusion
105 governing equation for the multilayered systems using the relative humidity which was later
106 used to determine the bond Interface Region Relative Humidity (IRRH) in different relative
107 humidity environment for different durations. This IRRH was correlated with bond fracture
108 energy to propose a bond deterioration model in moist environment (Ouyang and Wan 2009).

109 Even though there are already many existing studies on this topic, the clear distinct
110 mechanism of moisture effect on the material and the bond behavior is still not clear yet.
111 Great variation in the results and scarce explanations to such behavior prove that more
112 extensive experiments are necessary to investigate several different type of material, exposure
113 conditions, testing approaches etc. Therefore, the present experimental approach aims at
114 giving useful insight in long-term effect of moisture on the shear bond performance of FRP-
115 concrete composite under continuous and wet-dry cycles. As the durability of the bond is
116 highly dependent on the materials, mechanical and thermal test on the resins were also
117 carried out under such exposure conditions so as to determine its effects. This paper also
118 shade light on the effect of primer and substrate concrete strength on the bond failure
119 mechanism at the interface region by observing the failure patterns and degradation at the
120 interface due to moisture.

121 **EXPERIMENTAL PROGRAM**

122 The experimental study is divided into two different parts namely material test and bond test.
123 In the first part, effects of moisture on the mechanical properties of the constituent materials
124 such as primer, saturant and concrete were investigated and in the second part, the FRP-
125 concrete composite bond behavior was examined. Details of the specimen preparation,
126 material description, exposure conditions and experimental instrumentation are presented in
127 this section.

128 **Materials description**

129 ***Epoxy resin***

130 Two separate epoxy primers and an epoxy saturant were used in this study which are
131 collectively referred as epoxy resin. All three resins are room temperature curing epoxy
132 adhesives which are referred in the study as Epoxy-E, Epoxy-F and Epoxy-R. Epoxy-E is a
133 two component epoxy primer with base and hardener part mixed in the ratio of 4:1 by weight.
134 The base part is formulation of Bisphenol-A and Bisphenol-F type epoxy resin whereas
135 hardener is combination of polythiol and polyamines. This is a high modulus concrete
136 bonding primer mainly used for anchorage of steel rod in concrete, old-new concrete bonding
137 etc. Epoxy-F and Epoxy-R are also two component resins with base and hardener part mixed
138 in the ratio of 2:1 by weight. The base part is formulation of Bisphenol-A type epoxy resin
139 whereas hardener part consists of blends of polyamines. Epoxy-F is an epoxy primer whereas
140 Epoxy-R is the saturant for carbon fiber sheet supplied by the manufacturer. Higher viscosity
141 and presence of silica components makes it slightly different from Epoxy-F.

142 ***Continuous fiber sheet***

143 Unidirectional continuous fiber sheet made of carbon fiber is used having design thickness of
144 0.111 mm and Fiber Areal Weight (FAW) of 200 g/m². The tensile strength and modulus are
145 3400 N/mm² and 2.45×10⁵ N/mm² respectively.

146 ***Concrete***

147 Two different range of concrete strength were casted. They are referred as normal and high
148 strength concrete having mean cylindrical compressive strength as 39.4 MPa and 88.6 MPa
149 respectively. ASTM C39 (2004) was followed to obtain compressive strength of cylindrical
150 concrete specimens but the strength was calculated from the mean strength of one specimen
151 tested at different ages (0, 1, 2, 3, 4, 6 months for normal strength and 0, 3, 6 months for high
152 strength). 0 month refers to 28 days after casting and remaining durations were in reference
153 to this time. These concrete cylinders were subjected to environmental exposures only after 2
154 months together with other specimens.

155 **Specimen preparation**

156 ***Resin specimens***

157 Mechanical properties of the epoxy saturant and primers are determined by uniaxial tensile
158 and shear test. The tensile specimens were prepared in accordance with JIS K 7113 (1999) by
159 mixing base and hardener in the specified ratio to form dog-bone shaped specimens.
160 Dimensions of the specimen are shown in Fig. 1. The shear specimens were prepared
161 following JIS K 6850 (1995) by bonding two steel plates with a small quantity of epoxy
162 resins in between as shown in Fig. 2.

163 ***Bond specimens***

164 In order to investigate bond behavior, single lap shear bond specimens were prepared. The
165 concrete prisms along with cylinders were casted and cured for 28 days under moist condition
166 in the laboratory. The bonding surface of the concrete prism was grinded by disk sander to
167 remove the thin mortar layer and properly cleaned using compressed air to remove the dust
168 particles from the surface. The amount of surface preparation was visually judged and
169 attempt was made to make uniform surface roughness throughout the specimens. A thin
170 uniform layer of primer was applied on the surface and allowed to cure for 24 hours before
171 attaching carbon fiber sheet with necessary saturant as recommended by the manufacturer.
172 Both the resin and bond specimens were cured for more than a month before subjecting to
173 any kind of exposures. In a study conducted by Yang et al. (2008), 90% of the resin was
174 found to be cured in a day and more than 95% in a period of a week. Therefore, it is assumed
175 that a month long curing would be sufficient and the post-curing effect will not be substantial.
176 The details of the specimen are shown in Fig. 3. The specimen with Epoxy-E and Epoxy-F
177 primers together with Epoxy-R saturant are referred as Type-E and Type-F specimens
178 respectively. The bond length of 200mm was adopted. This bond length was adopted in
179 reference to the previous research by Dai (2003) in which the author found the effective bond
180 length varying from 50 mm to 100mm for the given stiffness value.

181 Exposure Conditions

182 All the specimens were subjected to two different environmental exposures apart from the
183 unexposed specimens. First group of specimens were subjected to continuous immersion into
184 a shallow tank containing tap water maintained at a constant temperature of 20 °C. Fig. 4
185 shows the arrangement of the tank with heaters, pump and temperature sensors to maintain
186 uniform temperature throughout the pool. After the required ageing period, specimens were
187 taken out from the water and tested immediately under wet conditions. The second set of

188 specimens were subjected to wet-dry cycles for a period of 3, 6, 12 and 18 months. There is
189 no standard for this kind of test so different duration of wet-dry cycles were adopted by past
190 researchers. Taukta et al. (2011) have considered 4 days of drying period and Dai et al.
191 (2010) have taken 4 days wetting and 3 days drying cycles. Similarly, in this study wet-dry
192 cycle include 7 days of immersion in water maintained at 20 °C and 4 days of drying inside
193 temperature control room at 20 °C. The humidity inside the control room was not
194 continuously monitored but initially it was in a range of 50-55%. The authors have
195 considered the specimens as wet and dry when the change in weights were less than 0.15% in
196 a consecutive day. It took 7 days of wetting and 4 days of drying in order to attain that criteria.
197 The remaining sets of unexposed specimens were put in ambient conditions inside the
198 laboratory. These specimens are considered as the control specimens for the study. At least 3
199 specimens were tested for controlled cases in contrast to 1 specimen for exposure case. The
200 presented results are the mean values of the identical specimens for the controlled case which
201 is referred here as 0 month. Summary of all the parameters along with the number of
202 specimens tested are presented in Table 3.

203 Test Procedures and Instrumentation

204 Tensile and shear test of the resin samples were conducted in universal testing machine
205 (UTM) at the loading rate of 2 mm/min and 1 mm/min respectively. The bond test was also
206 carried out in UTM with the arrangement illustrated in Fig. 5. Three long bolts were inserted
207 through the preset plastic pipes inside the concrete specimens and then fixed at the base of the
208 machine. The specimen was adjusted in position to make sure that the FRP-concrete bond
209 line is aligned with the center line of the upper loading grip. Strain gauges were attached on
210 some of the specimens at an interval of 20 mm on the bonded region to record the strain
211 behavior of the FRP sheet and Linear Variable Displacement Transducer (LVDT) was

212 positioned at the starting point of the bonding zone to measure the loaded end slip between
213 the concrete and FRP sheet. The loading speed of the upper grip was set as 0.2 mm/min.

214 The effect of exposures on the glass transition temperature (T_g) of the resin was determined
215 by differential scanning calorimetry (DSC) method following ASTM E1356 (2004). The
216 temperature range of measurement was -30 to 150 °C at a heating rate of 20 °C/min. T_g point
217 was chosen from the midpoint of the tangent between the extrapolated baseline before and
218 after transition.

219 **RESULTS AND DISCUSSION**

220 **Moisture absorption by epoxy resin specimens**

221 Moisture absorption by the epoxy resin is measured in each tensile specimen through
222 gravimetric means and expressed as the percentage increase in weight. Fig. 6 shows the
223 amount of moisture absorption by all three epoxy resins until 24 months duration. The
224 moisture absorbed at the end of 24 months by Epoxy-E, Epoxy-F and Epoxy-R were 1.98%,
225 2.63% and 2.50% respectively. Epoxy-F and Epoxy-R showed very resembling absorption
226 trend which could be due to presence of similar chemical components. From the figure, it can
227 be observed that the moisture diffusion was much quicker at the initial stages of immersion
228 which became more gradual after 6 months till 18 months. By the time it reaches 24 months,
229 the absorption curves became steadier suggesting the point of complete saturation. The
230 saturation time and the maximum amount of moisture absorption by resins are vastly
231 dependent on the epoxy resin types, its composition, curing period and immersion condition.
232 Variation of moisture absorption ranging from 0.86% to 4.06% was observed by Karbhari et
233 al. (2009) after 2 years of immersion in deionized water at 22.8°C when the authors
234 investigated 10 different resin types. Sciolti et al. (2010) reported even higher absorption of

235 water by the epoxy resins ranging from 4.7% to 8.2% after 24 weeks at 23 ± 2 °C. This kind of
236 variation in the moisture absorption characteristic makes it too difficult to generalize the
237 behavior of resin based on its moisture absorption property. Further, the nature of moisture
238 diffusion and absorption in the resin samples are different from the phenomenon involved in
239 FRP-concrete composite system as the resin layers are sandwiched between FRP sheet and
240 concrete. This makes diffusion process different and complex than only in case of resin
241 sample. Consequently, this absorption data is only used as a reference purpose to make a
242 comparison between different resins.

243 Effect of water on mechanical behavior of the resins

244 Fig. 7 shows the result of mechanical behavior of the three epoxy resins tested after
245 continuous exposure in water at various exposure durations. All the resins show fairly
246 consistent trend in response to immersion in water. Fig. 7(a) shows the response of tensile
247 strength with the exposure duration. The data seems to be scattered a bit thus making it
248 difficult to explain the clear tendency in response to the exposure duration but initial increase
249 in tensile strength can be observed in all three cases. Lau et al. (2009) also found out such
250 increase in tensile strength with the increase in moisture absorption at 23 °C when a
251 commercial two-component concrete bonding epoxy was examined but the reason for this
252 behavior was unknown. May et al. (2002) have explained this initial increase as the effect of
253 the toughening behavior of the resins due to water. Another possibility for such behavior
254 could be due to post-curing effect of the resin, but considering the curing period of more than
255 a month and the temperature of the water being only 20 °C, the post-curing phenomena may
256 not be the case here. It is also evident that the behavior shown by Epoxy-F and Epoxy-R
257 shows a great resemblance similar to the case of moisture absorption. The results indicate a
258 sudden drop of strength for Epoxy-F and Epoxy-R at the 18 month but the recovery of the

strength at the 24 month period suggests that it could only be the experimental scatter rather than the real deterioration. In overall, apart from small insignificant occasional reduction in the tensile strength, all three epoxy resins did not show any serious response to moisture. Among three resins, Epoxy-E seems to be most insensitive to moisture. As seen from the Fig. 7(b), similar to the tensile strength behavior, tensile modulus also showed variation at different durations. Even though there seems to be a small reduction, the variation indicates that there is no strong relationship of tensile modulus with the exposure duration therefore, all the immersion cases are averaged to see the overall response after immersion. It was found that in average, the modulus was reduced by 11, 11 and 4% respectively for Epoxy-E, Epoxy-F and Epoxy-R after exposure. Fig. 7(c) shows variation of shear strength at various exposure durations. The average reduction in shear strength was around 2, 16 and 14% for Epoxy-E, Epoxy-F and Epoxy-R respectively. As the tensile behavior, Epoxy-E seems to be less affected than the remaining other two resins which could possibly due to lower moisture absorption capacity. It is also interesting to observe that despite minimal effect on the tensile strength of the resins, the stress-strain curves changed to ductile nature with reduced stiffness after exposure thus increasing the strain at the failure. This behavior was observed in all three cases in both exposure conditions which are presented in Fig. 8. This increase in fracture strain at the failure is also reported by Petrie (2006) as one of the effects of water on the epoxy resin which can be recovered fully when dried.

Fig. 9 shows the effect of wet-dry cycles on the mechanical properties of the resin at different exposure durations. The overall nature on the mechanical behavior of the resins under wet-dry cycles are similar to the continuous exposure case. There was no reduction in tensile strength until 18 months period whereas slight reduction in tensile modulus was found as 4, 11 and 6% in average for Epoxy-E, Epoxy-F and Epoxy-R respectively. The shear strength was also decreased by 10 and 9% respectively in average for Epoxy-F and Epoxy-R whereas

284 no reduction for Epoxy-E. Comparison of results to the continuous immersion case does not
285 reveal much significant difference. Therefore, it can be concluded that the drying process
286 does not restore the original properties of the resins.

287 The above results indicate that there are some harmful effects of moisture on the mechanical
288 properties of resin but the extent of damage is not as severe as observed by few other
289 researchers such as Sciolti et al. (2010) and Yang et al. (2008) while comparing with the
290 similar exposure conditions. There exist a wide range of other previous studies showing
291 similar effects on the resin properties but with big variation in the results. This is mostly due
292 to different composition of the epoxy resins. Usually, the room temperature cured
293 commercial epoxy resins consist of two components namely base part and hardener part
294 which is the curing agent. The base part is mainly composition of Diglycidyl Ether of
295 Bisphenol-A (DGEBA) which is formed by reaction between Bisphenol A and
296 epichlorohydrin but the hardeners are blends of several different components such as amines,
297 amides, sulphide, thiols etc. This blending of different chemicals and addition of various
298 modifiers such as flexibilizers, tougheners, diluents, fillers, thixotropic agents etc. makes each
299 commercial epoxy with the unique characteristics. These modifications on the resin
300 properties are done to improve gel time, increase curing rates and achieve better resistance
301 against heat, chemicals and water. The properties of the resin could be adjusted based on the
302 user-intended application as a result of which durability related studies conducted by using
303 resins from different manufacturers may yield vastly different conclusions. But unfortunately,
304 information on the chemical composition of the resins are scarcely provided. Therefore more
305 specific studies need to be carried out providing such chemical information as well.

306 Effect of exposure on the glass transition temperature (T_g) of the resins

307 Fig. 10 shows the transition of epoxy resins over a range of temperature. The T_g is measured
308 at the midpoint of the temperature range bounded by the tangents of the two flat regions of
309 the heat flow curve. At the beginning, before subjecting to any sort of exposures, T_g were
310 measured as 71.2, 72.2 and 74.2 °C respectively for Epoxy-E, Epoxy-F and Epoxy-R. To
311 know the changes in T_g for each exposure condition, T_g were measured as a function of
312 exposure duration for each case. The results presented in Fig. 11 clearly indicate that the T_g
313 change is well under 5% over 24 months of exposure which is fairly insignificant. There are
314 some conflicting literatures on the effect of moisture on the T_g of the resin. Abanilla et al.
315 (2006), Wright (1981), Benzarti et al. (2011) all found significant decrease in T_g of the resins
316 caused due to absorption of moisture by the epoxy resins. Wright (1981) even showed the
317 linear relationship between decrease in T_g and the moisture content of the epoxy resin. The
318 author also proposed roughly decrease of T_g by 20 °C for each 1% water absorption. In
319 contrast to that, Choi and Douglas (2010) reported increase in T_g of epoxy-amine thermoset
320 resins after immersion in water at different temperatures. The reason for such behavior is
321 explained by increase in cross-link density due to additional curing at higher temperature. All
322 the above studies were conducted at the presence of moisture in the sample, but in this study,
323 samples were kept at room temperature for more than a month for drying purpose before
324 measuring the T_g to see the permanent effect of water on the resin. This process may have
325 restored the T_g back to the unexposed case.

326 Effect of water on compressive strength of concrete

327 As can be seen from the Fig. 12, change in compressive strength of the high strength concrete
328 seems to be insignificant over a period of 12 months whereas small reduction is noticeable in
329 case of normal strength for both continuous immersion and wet-dry cycles. It is believed that
330 the mechanical properties of the concrete decreases with increase in the moisture content.

331 Several previous researchers have studied the effect of moisture in the mechanical properties
332 of the concrete (Bazant and Prat 1988; Konvalinka 2002; Shoukry et al. 2011) and reported
333 decrease in the compressive strength with the degree of saturation. The reason for decrease is
334 due to intensified pore water pressure resulting into generation of micro cracks during
335 application of external forces. Comparatively, wet-dry case showed better performance due to
336 restoration of strength after drying. In case of high strength concrete, similar phenomenon
337 would occur but due to fewer and smaller voids, intensity of pore water pressure is much
338 lower but material resistance properties are much higher causing insignificant effect on the
339 strength.

340 **Effect of water on bond strength for the normal and high strength concrete**

341 Fig. 13 (a) and (b) show the effect of continuous immersion and wet-dry cycles in the bond
342 strength for Type-E and Type-F specimens with normal strength concrete. The bond strength
343 was calculated from the peak load obtained from the test. In response to the continuous
344 exposure, there is a slight different trend for Type-E and Type-F specimens. For Type-E,
345 bond strength along the exposure duration were slightly lower in a range between 5 to 12 %
346 than the non-immersion in most cases expect for the period between 6 to 12 months.
347 However, the increase is only significantly greater for the case of 12 months, which could be
348 the experimental scatter. For the Type-F case, the bond strengths after exposure were either
349 very similar or lesser than the non-immersion case. The reduction in bond strength can be
350 found up to the range of 1 to 12 %. Clearly, no specific trend of change in bond strength with
351 the immersion duration was found. But the observed reduction in bond strength for some
352 cases may be attributed to weakening of adhesion bond at the interface which is explained
353 along with the help of failure patterns at the later part. The results obtained from wet-dry
354 cycles also show reduction in bond strength ranging from 7 to 12 % for Type-E specimen and

355 1 to 3% for Type-F specimen except for the case of 12 months as similar to continuous
356 immersion case. In summary, we can see that there are some reductions in bond strength at
357 both exposure conditions but these reductions are not so significant and do not have any clear
358 relation with the exposure duration.

359 In contrast to the behavior shown by normal strength concrete specimens, both Type-E and
360 Type-F specimens showed greater loss in bond strength with the increase in immersion
361 duration for the high strength specimens. As seen in Fig. 13 (c), the maximum bond strength
362 reduced up to 32% after a year immersion into water. Similar decreasing trend was confirmed
363 for Type-F specimens with reduction of around 30% after a year of water immersion. It is
364 also interesting to note that even with the increase in the concrete strength, there is no
365 significant increase in the bond strength. Despite use of same resins and specimen preparation
366 procedure, the use of different strength concrete affected the bond durability. This brings into
367 focus at the interaction of bond between concrete and epoxy resin. There are two key
368 mechanisms which govern the bond between FRP composite and the concrete substrate. The
369 first is the mechanical bond which is the function of degree of surface roughness, while the
370 other one is the intermolecular forces or chemical bonds formed as a result of reaction
371 between epoxy components (mainly hydroxyl group) with the substrate concrete. Amount of
372 hydroxyl groups is dependent on the epoxy resins so use of same resin on both the surfaces
373 eliminates the possibility of making such difference thus making the mechanical bond as the
374 primary concern. Mechanical bond works by forming interlocking action formed by
375 penetration of resins into the pores, defects and surface roughness in concrete. Numerous
376 articles have been published on the effect of surface preparation on the bond strength (Al-
377 Tamimi et al. 2011; Toutanji and Ortiz 2001). Even though surface roughness is not the only
378 governing parameter, usually surface having higher roughness yields greater strength. High
379 strength concrete consists of closely packed cement grains and reduced amount of pores

380 compared to normal strength concrete which makes it denser and compact. The bonding area
381 and surface roughness also becomes lower in high strength concrete due to same reason.
382 Further, the surface roughness created by disk grinding method may not be sufficient to
383 create required roughness for the high strength concrete case which is evident by looking at
384 failure surface. The failure occurred mostly at the interface between concrete and primer in
385 contrast to the complete concrete cohesion failure in normal strength case suggesting
386 inadequate surface roughness. These reasons make the mechanical bond comparatively
387 ineffective than normal strength concrete case thus hindering the full utilization of high
388 strength concrete substrate. Above explanation can somehow help to understand the reason
389 for greater reduction in bond strength after exposure to water. Similarly, to the normal
390 strength concrete, there is a weakening of adhesion bond at the interface by the water but due
391 to ineffective mechanical bond in case of high strength concrete, the bond cannot be retained
392 resulting in premature failure thus lowering the bond strength. Here it should be noted that
393 better surface roughness in case of high strength concrete may yield different result for which
394 further investigations are required. But for now above presented theory is well supported by
395 observed failure patterns which are explained below.

396 **Effect of moisture on the failure modes for normal strength concrete**

397 The observation of failure surfaces gives good idea on the propagation of the crack path.
398 Even without any environmental exposures, there exist slight differences in failure surfaces
399 for Type-E and Type-F cases. Epoxy-E primer, being specifically used for concrete to
400 concrete bonding, showed greater adhesion with the concrete than Epoxy-F case. The
401 comparison of failed FRP sheets subjected to continuous immersion and wet-dry cycles at
402 different durations are presented in Fig. 14 (a) and (b). Careful observation indicates
403 difference in failure pattern after exposure in water for both cases. The failure before the

404 immersion was mostly at the thin concrete layer which later shifted to primer-concrete
405 interface region after immersion. Such shift in failure modes after interaction with water were
406 also observed by other researchers as well (Benzarti et al. 2011; Dai et al. 2010; Tuakta and
407 Büyüköztürk 2011). This kind of phenomenon is observed in both specimen types (Type-E
408 and Type-F) indicating some harmful effects of water on the bond at primer-concrete
409 interface. Although both resins and composite bond properties showed some signs of
410 deterioration under moisture conditions, their correlations between remains unclear since the
411 bond failure was at the interface region and therefore may need further investigations. But in
412 overall interface bond is clearly affected by moisture and shift in failure modes are evident.

413 The shift of failure to primer-concrete adhesion from concrete cohesion could have two
414 different possibilities. The first could be due to increase in shear strength of the substrate
415 concrete as a result of post-curing of the concrete thus making the adhesion layer more
416 vulnerable to failure. But if we consider the results obtained from the concrete cylinder test as
417 reference, then it clearly indicates that the strength is not further increased despite continuous
418 immersion in water for several months which denies the above hypothesis. This leads to
419 conclusion that the adhesion bond becomes weaker due to interaction of water molecules
420 with the epoxy-concrete bond. To understand more precisely, the bond mechanism at the
421 interface needs to be clarified.

422 In FRP-concrete bond system, among different layers and interfaces, the weakest interface is
423 often recognized as the resin-concrete interface by many past researchers. In case of wet
424 layup bonding, primer is usually applied on the concrete surface to enhance the bond. This
425 low-viscosity primer penetrates through voids and defects present on the concrete surface and
426 strengthens thin layer of concrete at the top. This layer is referred as the transition zone in this
427 paper which is a sandwiched layer between primer and concrete layer. This layer of concrete

428 at this transition zone behaves differently than the adjacent concrete which is confirmed by
429 observation of interface by optical microscopy under ultraviolet light illumination as shown
430 in Fig. 15 (Djouani et al. 2011).

431 At the interface, usually the shear strength of the structural adhesives are very high compared
432 to that of normal strength concrete (Tu and Kruger 1996) and transition zone is already
433 stronger than the adjacent concrete layer due to enhancement of strength by penetration of
434 resin. When adequate surface preparation on the substrate concrete is provided, the adhesion
435 bond at the primer and transition zone interface always remain intact making the concrete
436 adjacent to the transition zone as the weakest layer. Thus as a result, failure occurs at the
437 concrete side when there is no influence of environmental exposures.

438 When this bonding system is introduced to wet environments, the moisture diffuses through
439 various different ways such as through concrete pores, adhesives, cracks or defects etc. and
440 reaches the interface region. This moisture at the interface could interact with specific
441 hydrophylic functional groups, such as hydroxyl or amine in epoxy resin and disrupt the
442 hydrogen bond (Bellenger et al. 1989). The evidence of such breakage of hydrogen bond due
443 to interaction with water were observed by some researchers (Lefebvre et al. 1991; Zhou and
444 Lucas 1999). Due to high porosity of concrete the moisture easily gains access to the
445 transition zone and the primer-transition zone interface where it combines with some of the
446 epoxy components (hydroxyl groups) thus partially destroying the adhesion bond. Due to
447 destruction of adhesion bond, adhesion strength at the primer-transition zone interface
448 becomes lower than the cohesion strength of concrete and failure tends to propagate along the
449 interface of primer and transition zone but due to presence of mechanical interlocking action
450 induced by surface roughness, the crack propagates towards the transition zone at some
451 locations thus causing mixed mode of failure. The visualization of bond failure mechanism

452 before and after the exposure is schematically demonstrated in Fig. 16 (a) for the normal
453 strength concrete. Similar phenomenon can explain the case of wet-dry exposure in which
454 water molecules destroy the adhesion bond at the primer-transition zone interface which
455 cannot be restored by the drying process (Tuakta and Büyüköztürk 2011).

456 **Effect of moisture on the failure mode for high strength concrete**

457 The failure observed in high strength concrete is little different from that of normal strength
458 case which is mainly due to surface condition of substrate and bonding mechanism. As
459 explained earlier, the dense nature of high strength concrete results in lesser voids due to
460 which primer does not penetrate deeply inside the concrete thus forming only a thin and week
461 transition layer as demonstrated in Fig. 16 (b). This reason makes interface between primer
462 and transition zone the weakest, causing propagation of cracks along the interface region.
463 However due to effect of thin transition zone, the crack occasionally propagates along the
464 transition zone creating mixed mode of failure in case of unexposed specimens. After
465 subjecting the specimens to water, the water molecules destroy the adhesion bond at the
466 primer-transition zone interface similarly as in case of normal strength case but unlike in
467 normal strength concrete case, mechanical bond is weaker due to much smoother surface thus
468 resulting into complete adhesion failure at the interface as shown in Fig. 14 (c).

469 **Effects of moisture on bond stress-slip behavior and fracture energy**

470 Bond stress-slip relation is the most important interface law which determines the overall
471 performance of the bonded members (Ueda and Dai 2004). The experimental local bond
472 stress-slip curves were obtained by integrating the strains measured on the surface of the FRP
473 composites with the interval of 20 mm along the bonded region (Dai 2003). Fig. 17 show
474 experimentally observed local bond stress-slip curves for normal and high strength concrete
475 specimens at different stages of exposures. The curves are plotted for the location of 30mm

476 from the starting point of the bonded end. Like the bond strength behavior, these local bond
477 stress-slip curves does not show clear distinct trend between non-immersion and immersion
478 cases for normal strength concrete for both exposure cases. In contrast to that greater
479 reduction in bond strength for high strength cases are well reflected in the local bond stress-
480 slip behavior. Clearly significant reductions in peak bond stresses and the overall change in
481 the nature of the curve can be observed after 12 months of immersion period.

482 The area under bond stress-slip curve gives the interfacial fracture energy which is the total
483 amount of energy consumed during the propagation of cracks. This fracture energy usually
484 governs the ultimate bond strength if the sufficient bond length is provided. Therefore, the
485 average fracture energy is calculated from average area of the experimental local bond stress-
486 slip curves at different locations before and after exposure. High strength cases showed
487 significant reductions in average fracture energy than for the normal strength specimens. For
488 the high strength case, the average fracture energy was reduced from 0.98 and 1.25 N/mm to
489 0.42 and 0.40 N/mm for Type-E and Type-F specimens after 12 months of exposure. These
490 results indicate some degradation of bond properties at the interface mainly caused due to
491 water as explained earlier. Further, significant loss of bond strength in high strength case after
492 the exposure is well supported by observed reduction in fracture energy.

493 The relationship between average fracture energy and exposure duration for normal strength
494 concrete specimens are shown in Fig. 18. In contrast to the behavior for the high strength
495 concrete, there is no distinct significant loss in average fracture energy at both exposure
496 conditions along the entire period. Initially, the average fracture energy were 0.56 and 0.84
497 N/mm for Type-E and Type-F specimens respectively which changed insignificantly to 0.52
498 and 0.82 N/mm after 24 months of continuous exposure. Even after 18 months wet-dry cycles,
499 the values were 0.59 and 0.81 N/mm for Type-E and Type-F respectively.

500 CONCLUSIONS

501 Durability of FRP-concrete bond and its constituent properties under the moisture conditions
502 are investigated for duration up to 24 months and following conclusions are drawn out:

- 503 1. Small reductions in mechanical properties of resins (modulus and shear strength) were
504 observed for both continuous immersion and wet-dry cycle case. However, original
505 properties cannot be fully restored even after drying process signifying permanent
506 damages due to such exposures. The effect of water caused more ductile behavior of
507 resins with increase in elongation at the failure.
- 508 2. The use of relatively high modulus primer (Epoxy-E) was proved to be good for this
509 FRP composite system. Such epoxy primers can be used as long as it is suitable for
510 concrete applications. However, durability needs to be confirmed before use.
- 511 3. Significant reduction on bond strengths was observed for high strength concrete
512 specimens than the normal strength case after exposed to continuous water immersion.
513 The durability of the bond strength was found highly dependent on the substrate
514 concrete and its surface roughness. Higher reduction in bond strength in high strength
515 concrete was attributed to weaker mechanical interlocking action due to relatively
516 smoother surface. Therefore, in case of high strength concrete, it is recommended to
517 use alternative method of surface preparation such as sandblast or water-jet to ensure
518 better bonding surface. However, durability of bond under different methods of
519 surface preparation needs to be further investigated.
- 520 4. The failure modes were changed after exposure in water for both normal and high
521 strength case. For the normal strength concrete, it shifted from a complete concrete
522 cohesion failure to a primer-concrete mixed type failure. Whereas in case of high

523 strength concrete, failure shifted from primer-concrete mixed failure to complete
524 adhesion failure due to relatively weaker mechanical interlocking action.

525 5. The effect of water was observed in the local bond stress-slip behavior. The interfacial
526 fracture energy is calculated before and after exposure to water. Comparison of result
527 show greater reduction in fracture energy and peak bond stress in the case of high
528 strength concrete subjected to 12 months of exposure than the normal strength. This
529 reduction in fracture energy corresponds well with the decrease in the bond strength.

530 **Acknowledgements**

531 The authors would like to acknowledge contributions made by Mr. Atsuya Komori and Mr.
532 Atushi Kitami for this research. The authors are also grateful to Nippon Steel and Materials
533 Co., Ltd., for providing necessary epoxy resins and carbon fiber sheet for this study. This
534 study was conducted with the financial aid provided by the Grant-in-Aid for Scientific
535 Research (A) No.22246058.

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- 625
- 626

Table 1 Summary of existing results showing mechanical and thermal effect on the epoxy resins after environmental exposures

Authors	Resin information	Exposure condition/duration	Property Changes		
			Reduction in mechanical properties (%)		T_g
			Tensile	Modulus	
Sciolti et al. (2010)	Two-component commercial epoxy primer	23 °C in water/ 27 wks	64	67	Reduction by 20 °C
	Two-component commercial adhesive	23 °C in water/ 29 wks	46	59	Reduction by 3 °C
	Two-component commercial putty	23 °C in water/ 31 wks	66	83	Reduction by 18 °C
Frigione et al. (2006)	Bisphenolic epoxy resin (MW<700)	23±1 °C in water/ 19 wks	9	17	Reduction by 3 °C
	Bisphenolic epoxy resin 66% inorganic filler	23±1 °C in water/ 38 wks	12	36	Increase by 3 °C
	Bisphenolic epoxy resin 49% of inorganic filler	23±1 °C in water/ 38 wks	18	21	Increase by 2 °C
Benzarti et al. (2011)	Bisphenolic epoxy resin and Polyamide hardener	40°C and 95% RH/ 265d	73	73	Reduction by 6-30 °C
	Bisphenolic epoxy resin and amine hardener	40°C and 95% RH/ 206 d	47	31	Increase by 20°C
Yang et al. (2008)	Bisphenolic epoxy resin and aliphatic amine	23°C in deionized water/ 24 m	47	34	Reduction by 5%
		37.8°C in deionized water/ 24 m	44	40	Insignificant
		60°C in deionized water/ 24 m	69	68	Insignificant
		23°C in salt solution/ 24 m	36	30	Reduction by 4%
		23°C in alkali solution/ 24 m	53	50	Reduction by 5%
Au (2005)	Bisphenolic epoxy resin and amine hardener	23°C in deionized water/ 4 wks	13	19	Reduction by 6 °C
		50°C in deionized water/ 4 wks	28	37	Reduction by 3 °C
	Two-component epoxy resin and amine hardener	23°C in deionized water/ 8 wks	22	15	Reduction by 7 °C
		50°C in deionized water/ 8 wks	10	10	Reduction by 3 °C

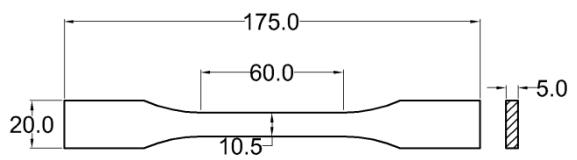


Fig. 1 Dumbbell shaped specimen for tensile test

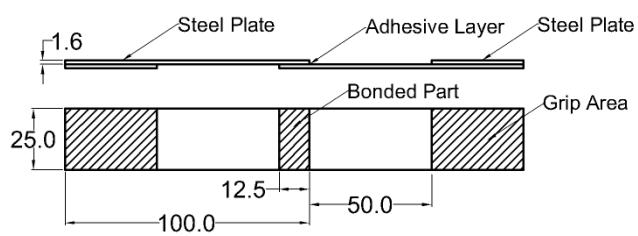


Fig. 2 Adhesively bonded metal specimen for shear test

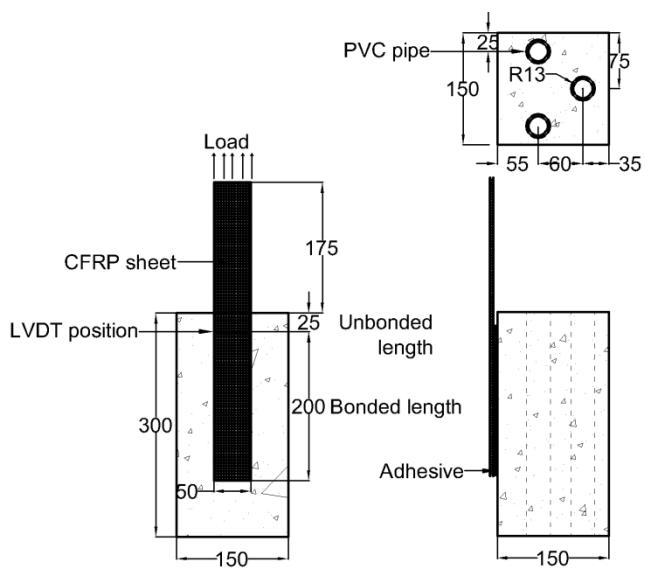


Fig. 3 Schematic of the bond specimen for single lap shear test

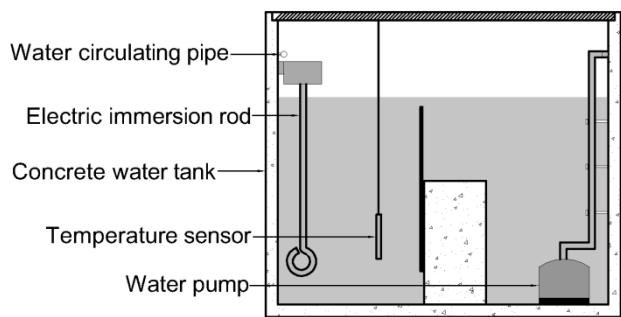


Fig. 4 Exposure tank

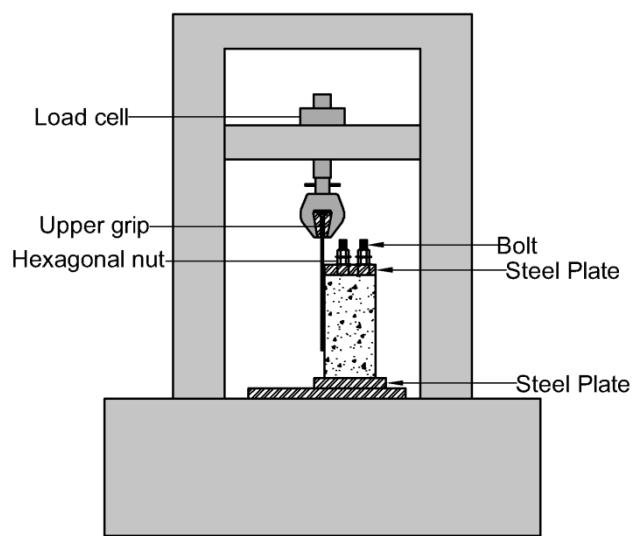


Fig. 5 Arrangement for the bond test

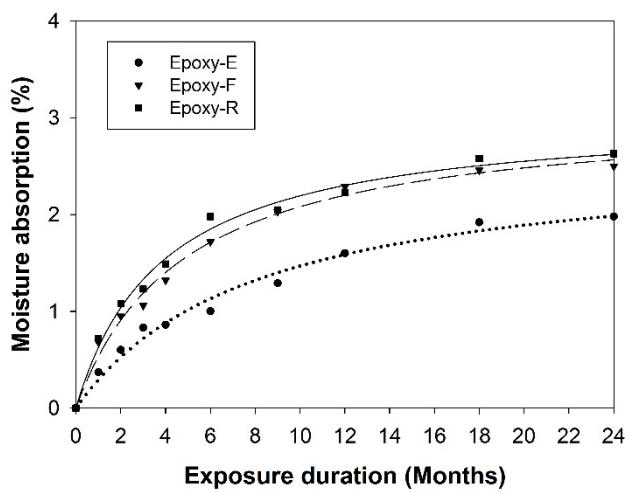
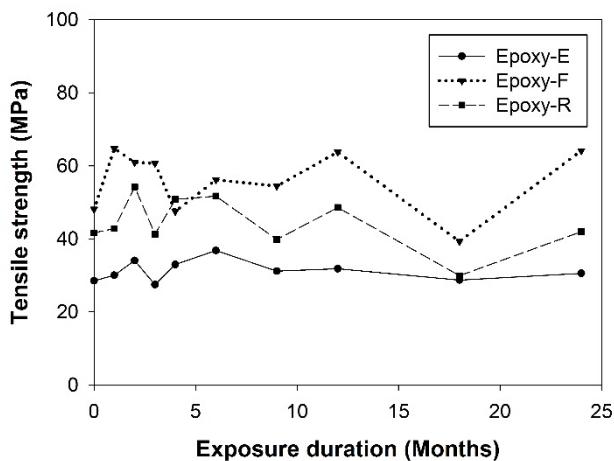
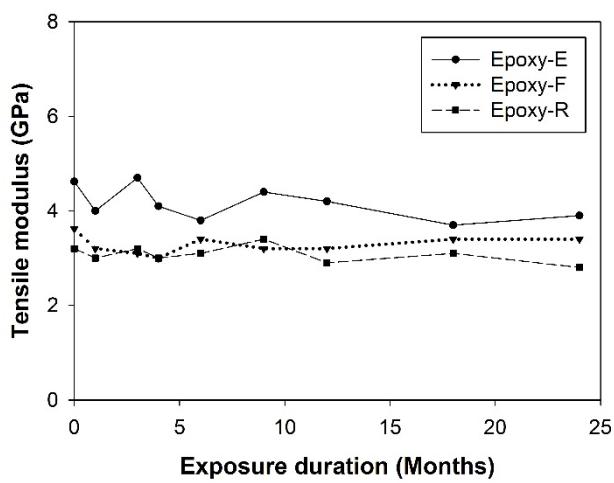


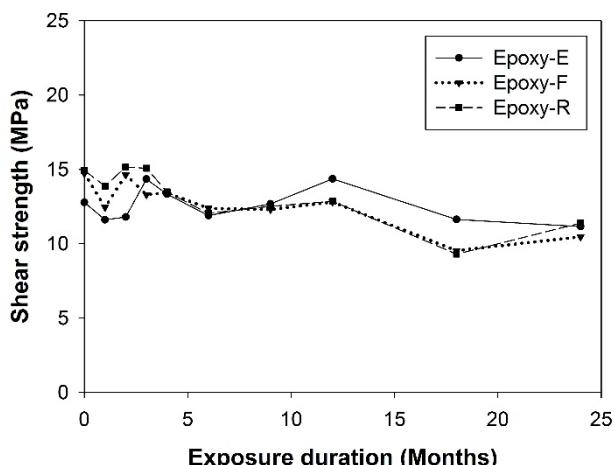
Fig. 6 Moisture absorption by epoxy resin specimens



(a)



(b)



(c)

Fig. 7 Mechanical properties of the resin after continuous exposure: (a) Tensile strength; (b) Tensile modulus; and (c) Shear strength

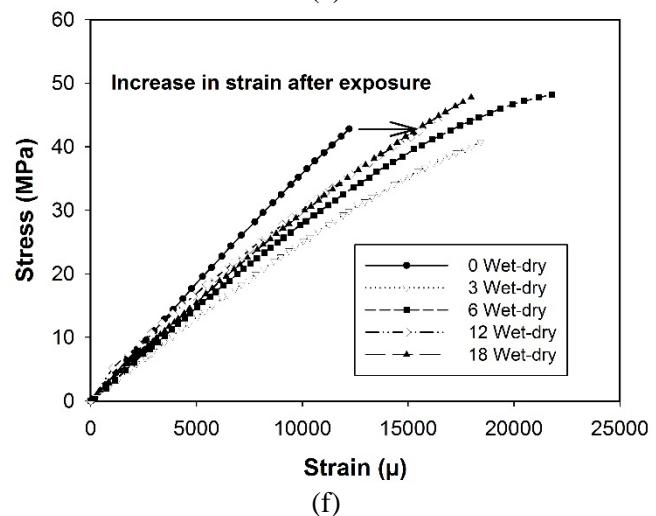
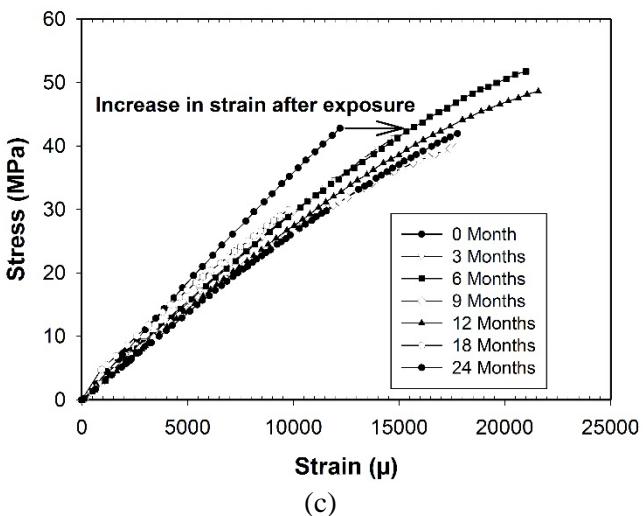
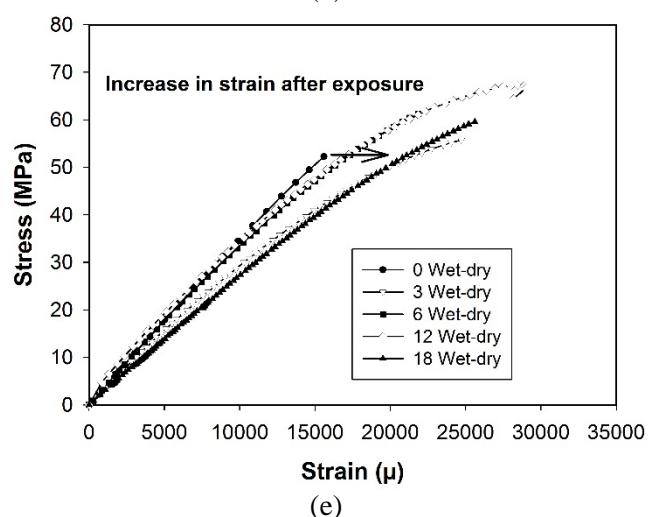
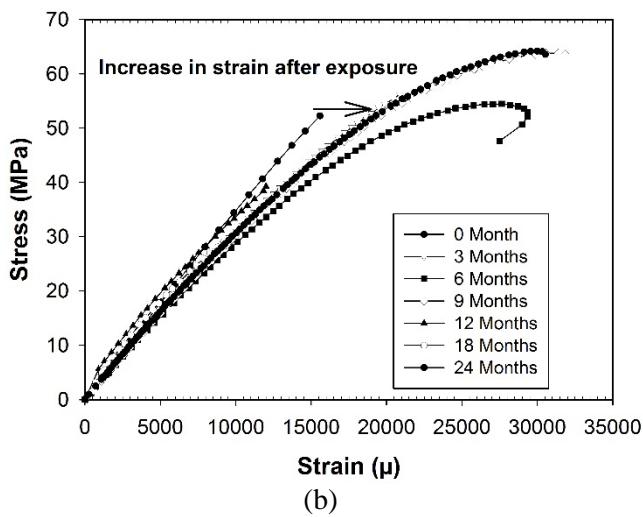
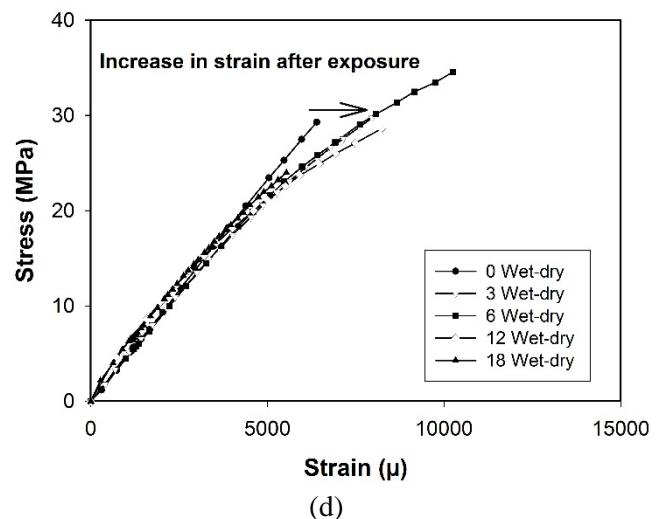
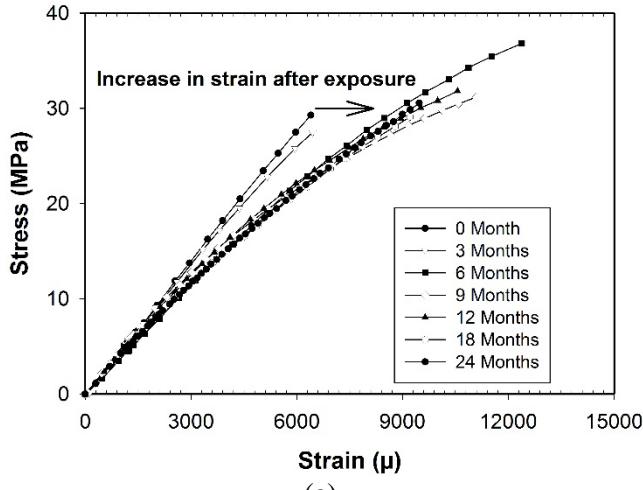
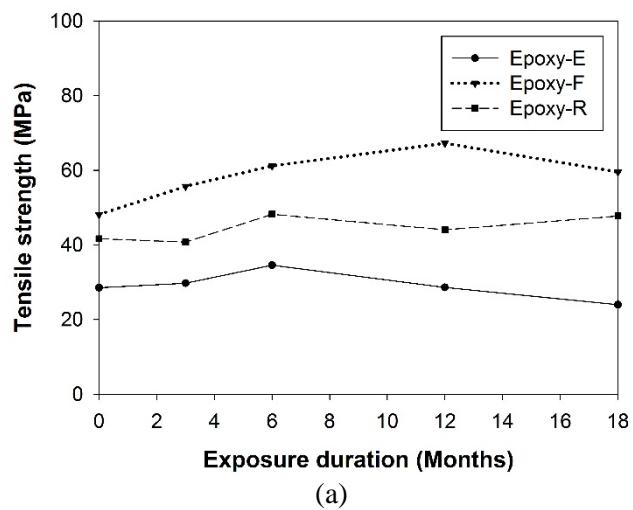
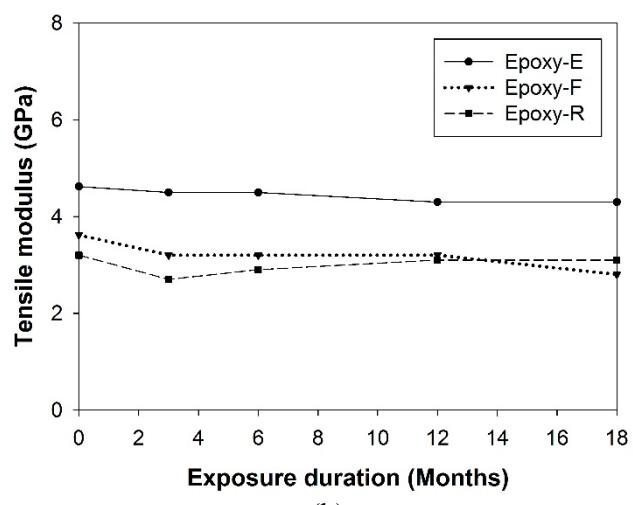


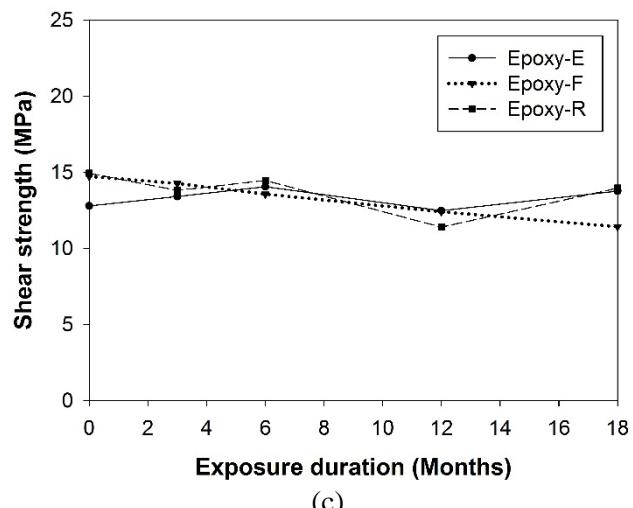
Fig. 8 Stress-strain curves for resins at different exposures: (a) Epoxy-E (Continuous immersion); (b) Epoxy-F (Continuous immersion); (c) Epoxy-R (Continuous immersion); (d) Epoxy-E (Wet-dry cycles); (e) Epoxy-F (Wet-dry cycles); and (f) Epoxy-R (Wet-dry cycles)



(a)



(b)



(c)

Fig. 9 Mechanical properties of the resin after wet-dry cycles: (a) Tensile strength; (b) Tensile modulus; and (c) Shear strength

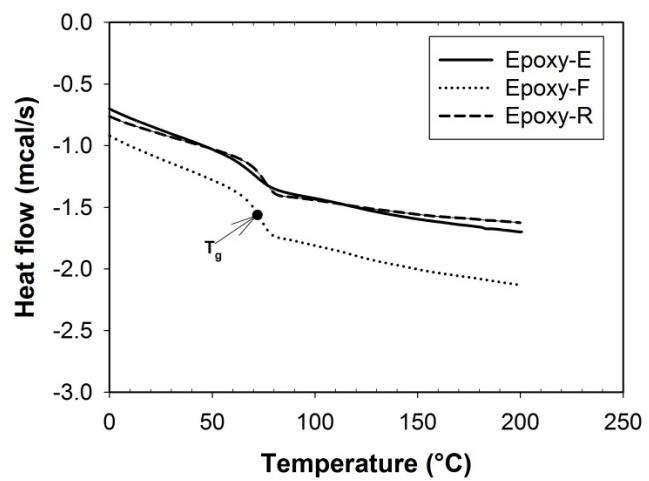


Fig. 10 Heat flow curves of epoxy resins before exposure

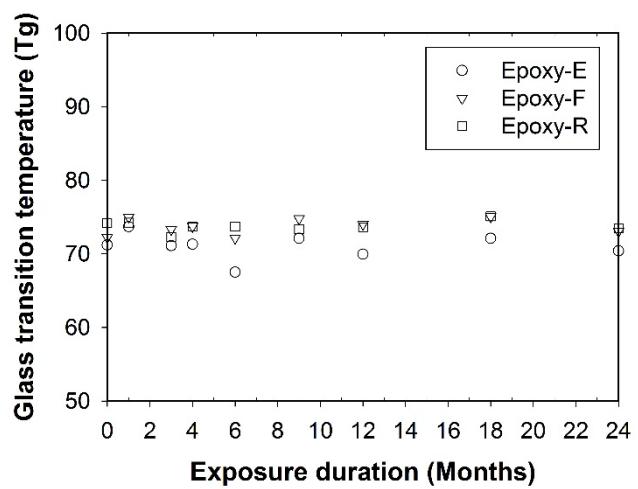


Fig. 11 Effect of continuous exposure on T_g of the resins after drying for a day

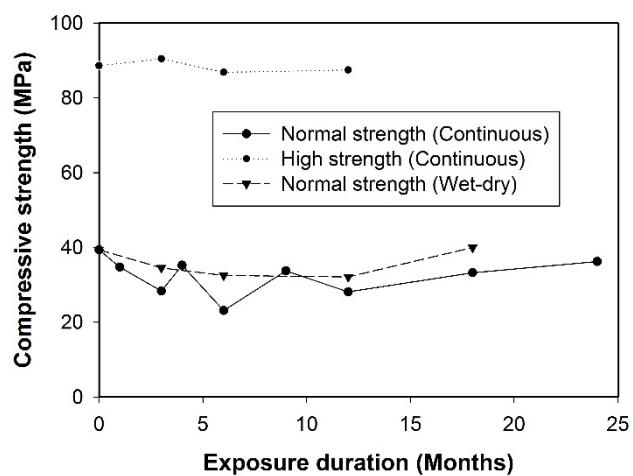
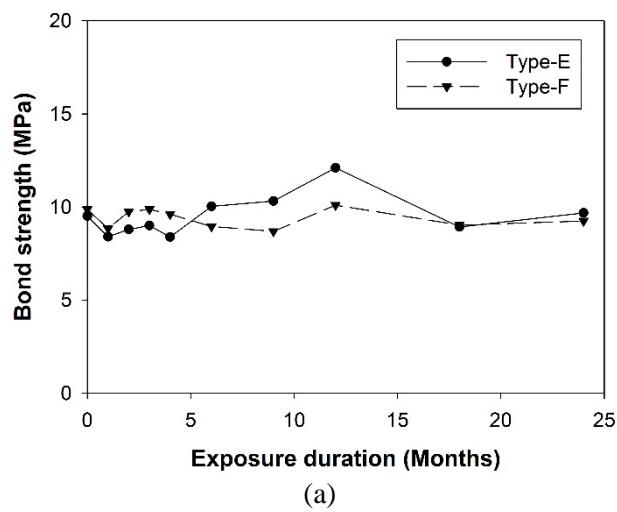
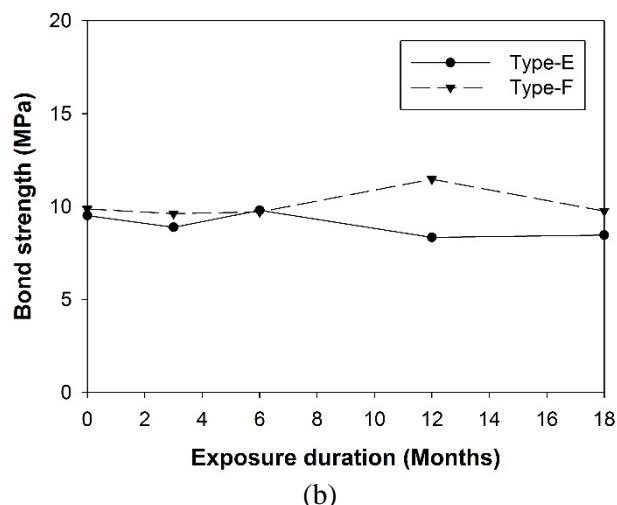


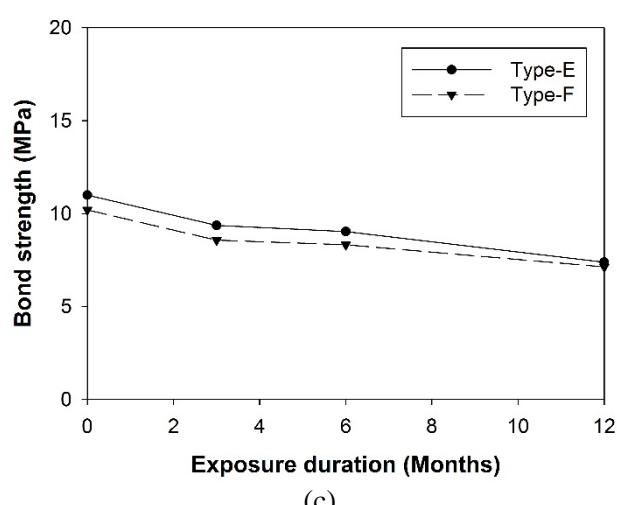
Fig. 12 Effects of exposure on concrete compression strength



(a)



(b)



(c)

Fig. 13 Effect of different exposures on bond strength: (a) continuous immersion and normal strength; (b) wet-dry cycles and normal strength; and (c) continuous immersion and high strength

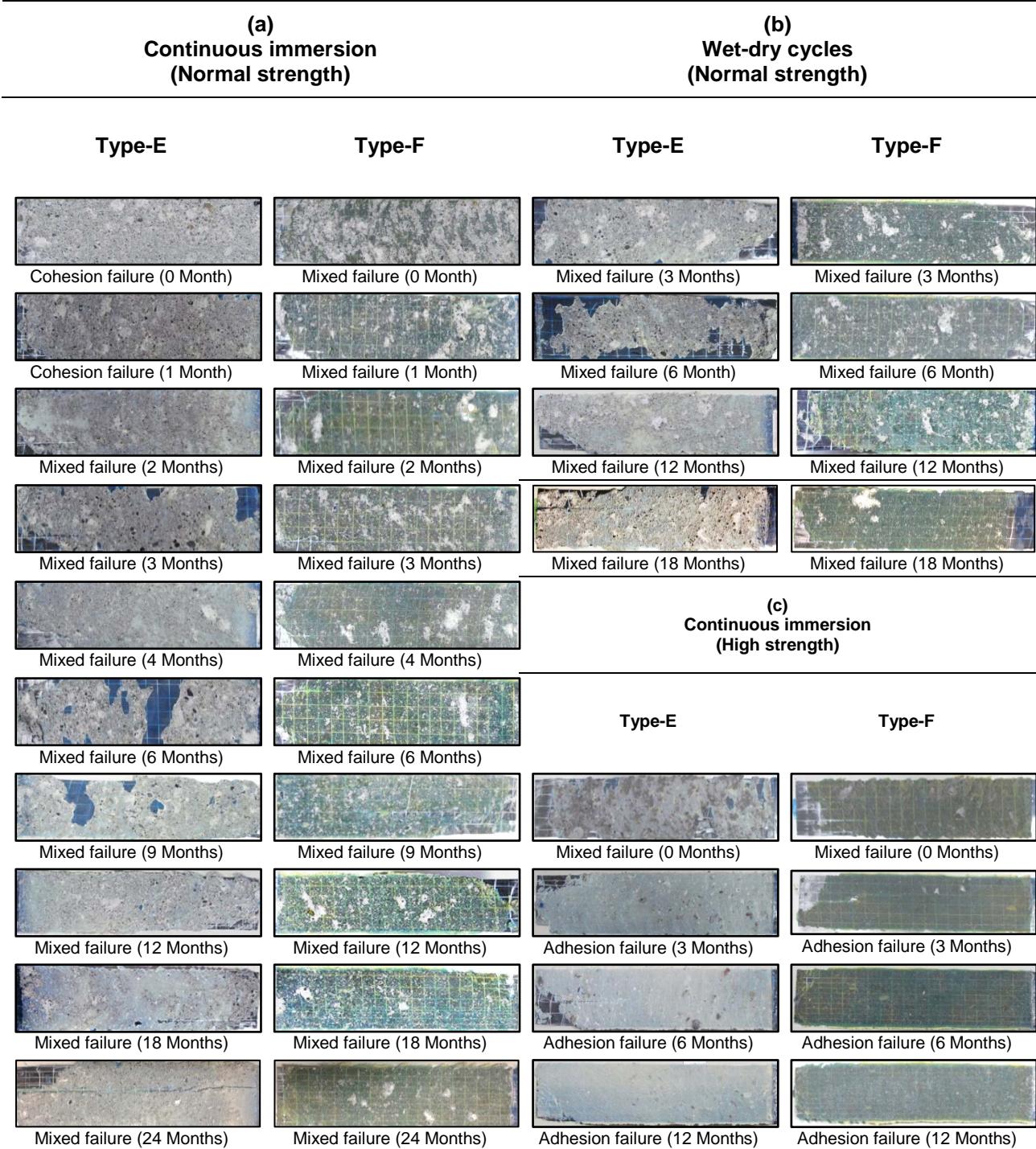


Fig. 14 Comparison of failure surfaces at various exposure conditions (a) continuous immersion for normal strength; (b) wet-dry cycles for normal strength; and (c) continuous immersion for high strength

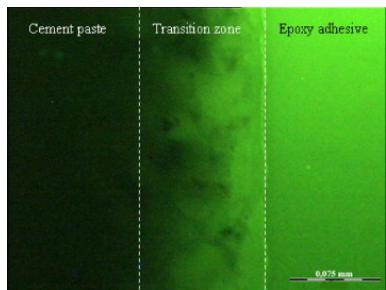


Fig. 15 Hardened cement paste-epoxy interface image obtained by optical microscopy under UV illumination (Djouani et al. 2011)

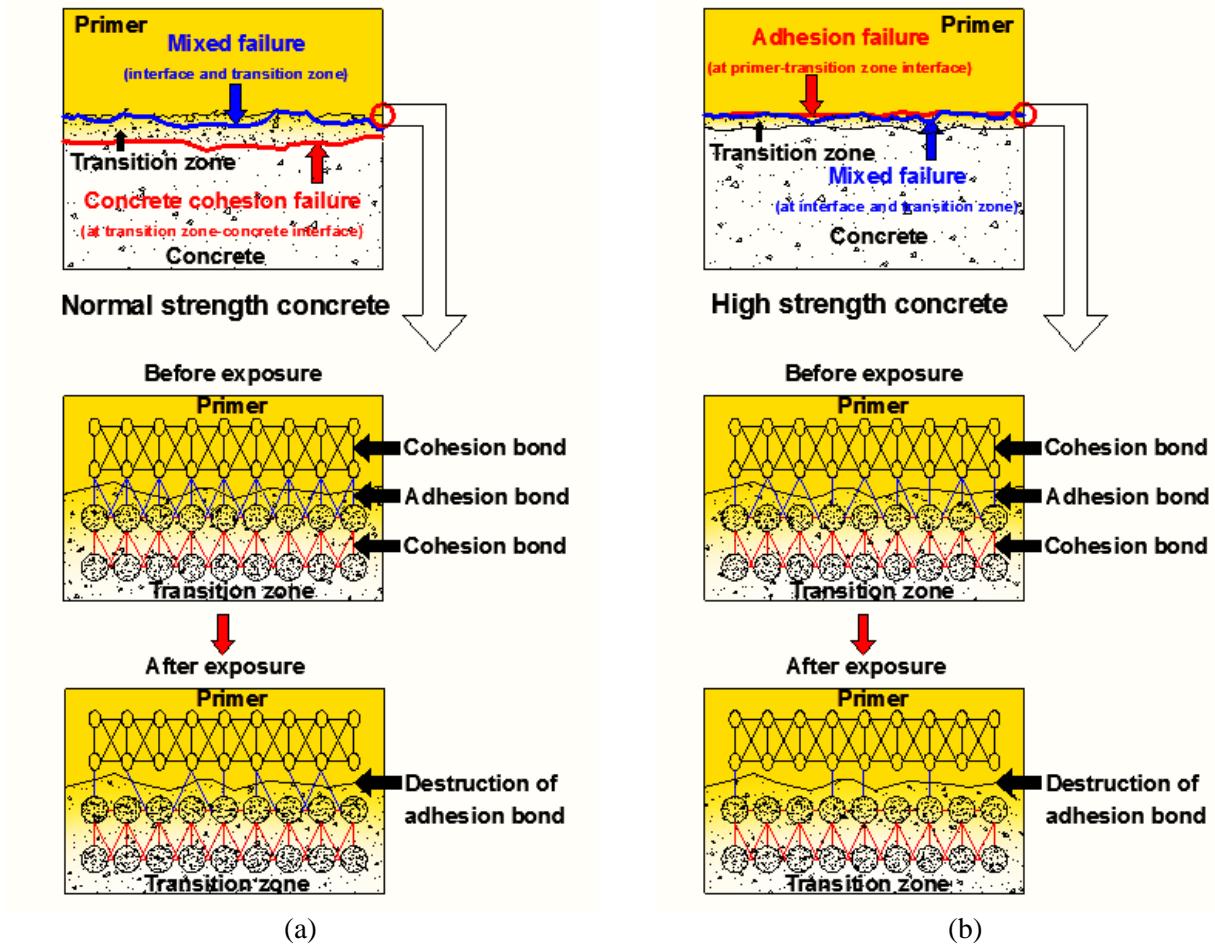


Fig. 16 Visualization of bond failure mechanism before and after exposure in (a) Normal strength concrete; and (b) High strength concrete

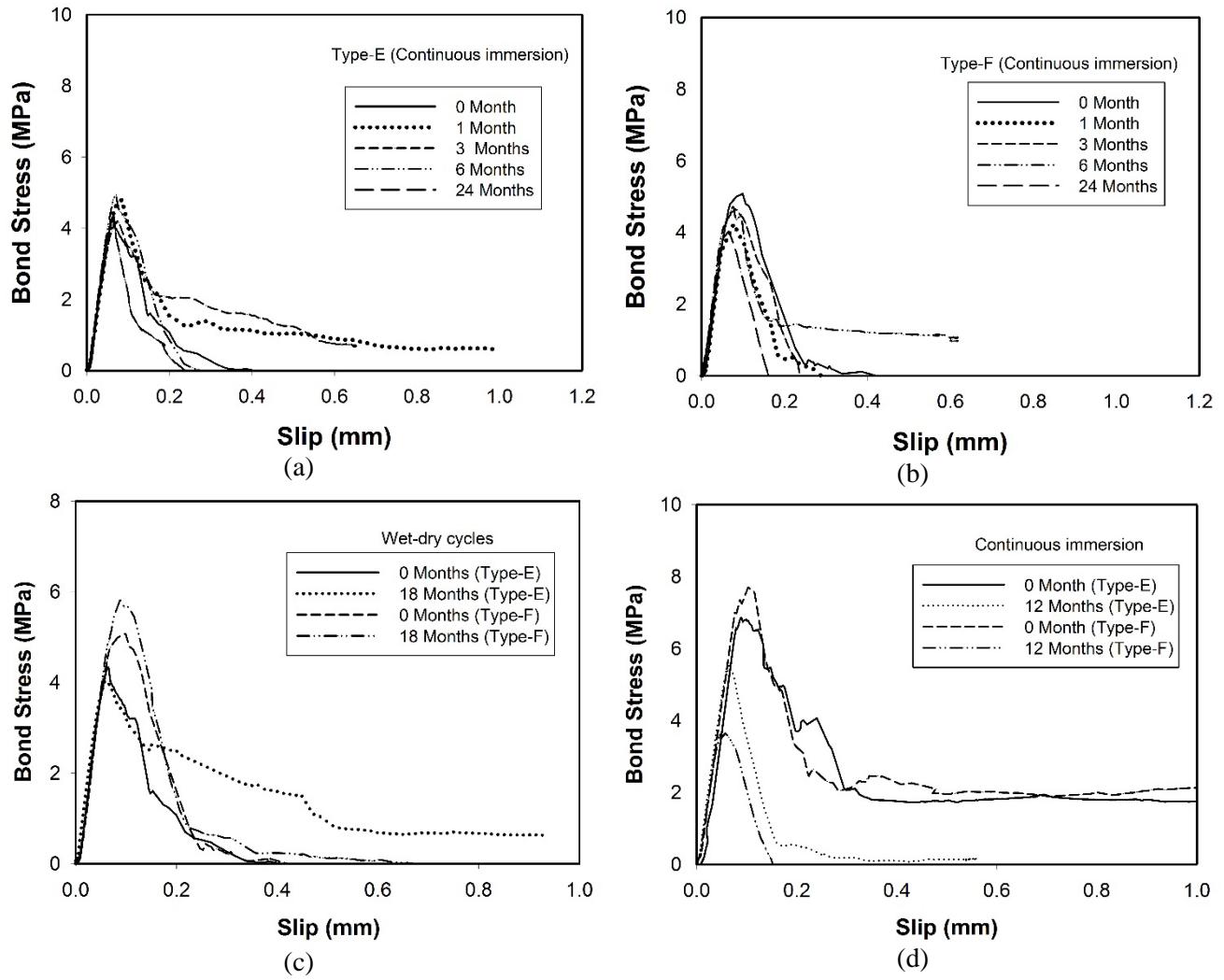


Fig. 17 Effect of moisture on local bond stress-slip curves at the same location for (a) normal strength concrete and continuous immersion (Type-E); (b) normal strength concrete and continuous immersion (Type-F); (c) normal strength concrete and wet-dry cycles; and (d) high strength concrete and continuous immersion

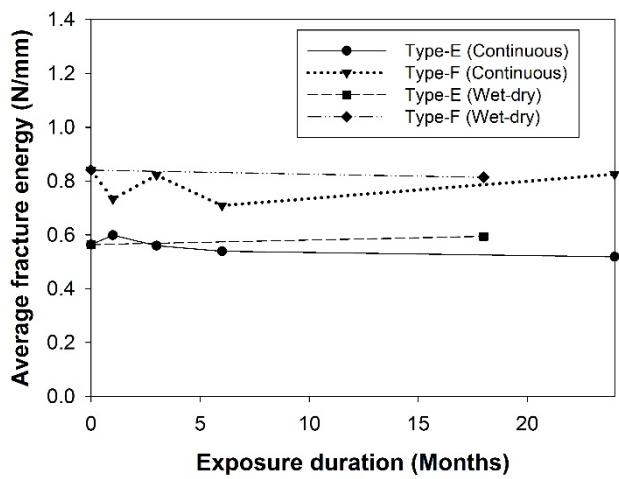


Fig. 18 Effect of moisture on average fracture energy for normal strength concrete

Table 2 Summary of the existing results for FRP-concrete bond after environmental exposures

Authors	FRP system	Exposure conditions/duration	Test	Results		Failure mode
				Bond strength		
Au (2005)	CFRP plate	23 and 50 °C in deionized water/ 2-8 wks	Peel test	60% decrease in peel fracture toughness	Epoxy/Concrete Interface separation	Epoxy/Concrete Interface separation
			Shear test	10, 50% decrease in shear fracture toughness	Epoxy/Concrete Interface separation	
Dai et al. (2010)	CFRP strand sheet with 3 different epoxies	4 days wet at 60°C sea water and 3 days dry/ 8-24 m	4 point bending	Epoxy-I: No change; Epoxy-II 25% increase in flexural strength; Epoxy-III 20% decrease in flexural strength	Primer/concrete interface separation	
Choi et al. (2012)	CFRP sheet with 3 different epoxies	30 , 40, 50, 60 °C Immersion/6-18 m	3 point bending	10-60% decrease in flexural strength for 3 epoxy system; 15% increase in flexural strength for 1 epoxy system	Adhesive failure	Inter-laminar composite failure
	CFRP plate			30, 40, 50, 60% decrease in flexural strength	Inter-laminar composite failure	
Benzarti et al. (2011)	CFRP sheet and plate	40°C, 95% RH/9-353 d	Shear test	10-15% increase in shear strength	Adhesive failure/interface	Concrete cohesive/mixed failure
	CFRP sheet and plate		Pull-off test	Decrease in pull-off strength up to 60% for plate system; increase in strength up to 25% for sheet system	Concrete cohesive/mixed failure	
Toutanji et al. (1997)	CFRP sheet with 3 different epoxies	300 WD cycles (4 hrs wet and 2 hrs dry)	4 point bending	Decrease in flexural strength 5-30%	-	
Karbhari et al. (1997)	CFRP sheet with different epoxies	Water at ambient temperature/ 120 d	4 point bending	Decrease in flexural strength 10-30%	-	

Table 3 Summary of parameters along with number of specimens tested for the study

Exposure duration (months)	Continuous immersion						Wet-dry cycles					
	Resin (Epoxy-E/F/R)	Concrete		Bond (Type-E/F)		Resin (Epoxy-E/F/R)	Concrete		Bond (Type-E/F)			
		Tensile Shear	T_g	Compression			Shear		Tensile	Shear	Compression	Shear
				Normal	High		Normal	High			Normal	Normal
0	6	6	3	6	3	6	6	3	-	-	-	-
1	1	1	1	1	-	1	1	-	-	-	-	-
2	1	1	1	1	-	1	1	-	-	-	-	-
3	1	1	1	1	1	1	1	1	1	1	1	1
4	1	1	1	1	-	1	1	-	-	-	-	-
6	1	1	1	1	1	1	1	1	1	1	1	1
9	1	1	1	1	-	1	1	-	-	-	-	-
12	1	1	1	1	1	1	1	1	1	1	1	1
18	1	1	1	1	-	1	1	-	1	1	1	1
24	1	1	1	1	-	1	1	-	-	-	-	-