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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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Abstract:

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

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

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/m^2. The tensile strength and modulus are

145   3400 N/mm^2 and 2.45×10^5 N/mm^2 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.

RESULTS AND DISCUSSION

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

259   strength at the 24 month period suggests that it could only be the experimental scatter rather

260   than the real deterioration. In overall, apart from small insignificant occasional reduction in

261   the tensile strength, all three epoxy resins did not show any serious response to moisture.

262   Among three resins, Epoxy-E seems to be most insensitive to moisture. As seen from the Fig.

263   7(b), similar to the tensile strength behavior, tensile modulus also showed variation at

264   different durations. Even though there seems to be a small reduction, the variation indicates

265   that there is no strong relationship of tensile modulus with the exposure duration therefore, all

266   the immersion cases are averaged to see the overall response after immersion. In was found

267   that in average, the modulus was reduced by 11, 11 and 4% respectively for Epoxy-E, Epoxy-

268   F and Epoxy-R after exposure. Fig. 7(c) shows variation of shear strength at various exposure

269   durations. The average reduction in shear strength was around 2, 16 and 14% for Epoxy-E,

270   Epoxy-F and Epoxy-R respectively. As the tensile behavior, Epoxy-E seems to be less

271   affected than the remaining other two resins which could possibly due to lower moisture

272   absorption capacity. It is also interesting to observe that despite minimal effect on the tensile

273   strength of the resins, the stress-strain curves changed to ductile nature with reduced stiffness

274   after exposure thus increasing the strain at the failure. This behavior was observed in all three

275   cases in both exposure conditions which are presented in Fig. 8. This increase in fracture

276   strain at the failure is also reported by Petrie (2006) as one of the effects of water on the

277   epoxy resin which can be recovered fully when dried.

278   Fig. 9 shows the effect of wet-dry cycles on the mechanical properties of the resin at different

279   exposure durations. The overall nature on the mechanical behavior of the resins under wet-

280   dry cycles are similar to the continuous exposure case. There was no reduction in tensile

281   strength until 18 months period whereas slight reduction in tensile modulus was found as 4,

282   11 and 6% in average for Epoxy-E, Epoxy-F and Epoxy-R respectively. The shear strength

283   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 flexiblizers, 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 Tg 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 expect 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 hydrophyllic 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 it 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 relative 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.

Acknowledgements

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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	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
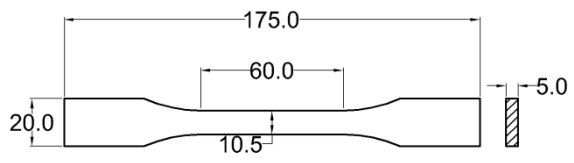
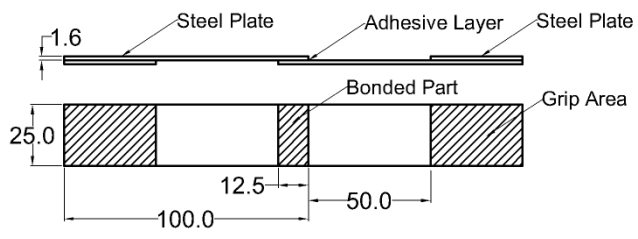**Fig. 1** Dumbbell shaped specimen for tensile test

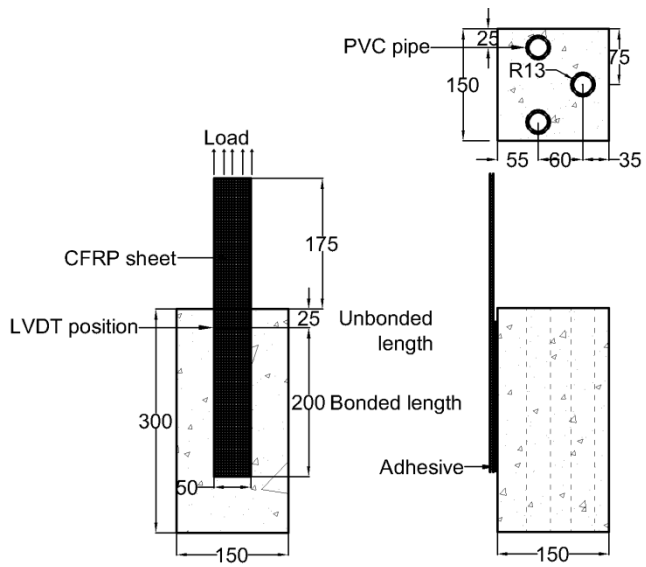**Fig. 2** Adhesively bonded metal specimen for shear test

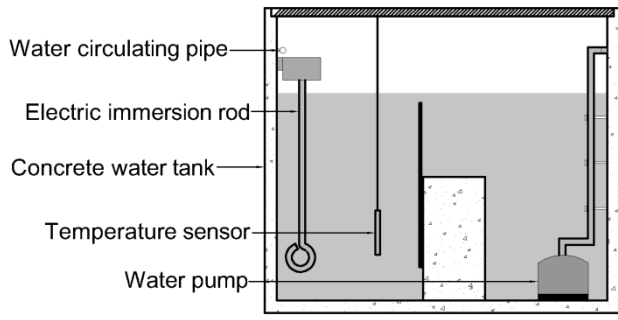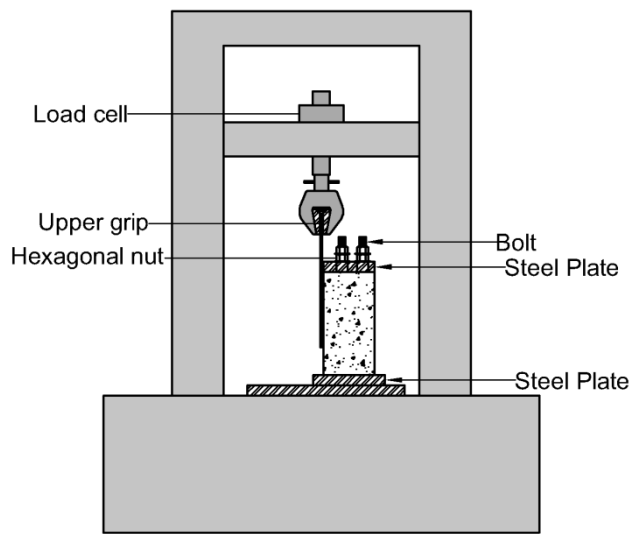**Fig. 3** Schematic of the bond specimen for single lap shear test

Fig. 4 Exposure tank
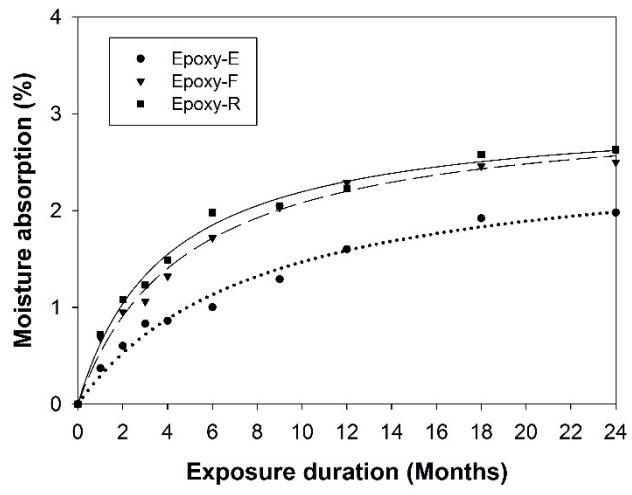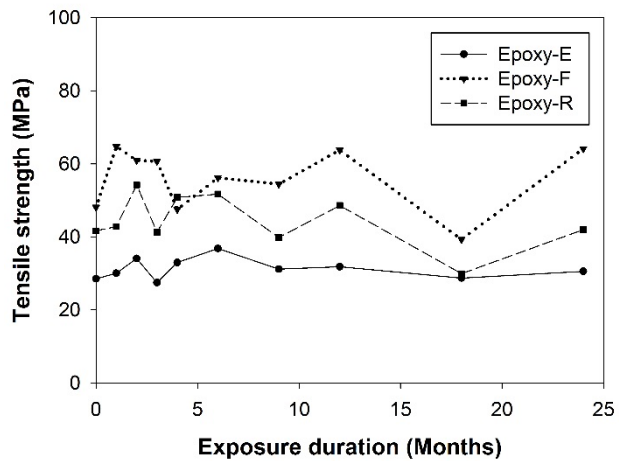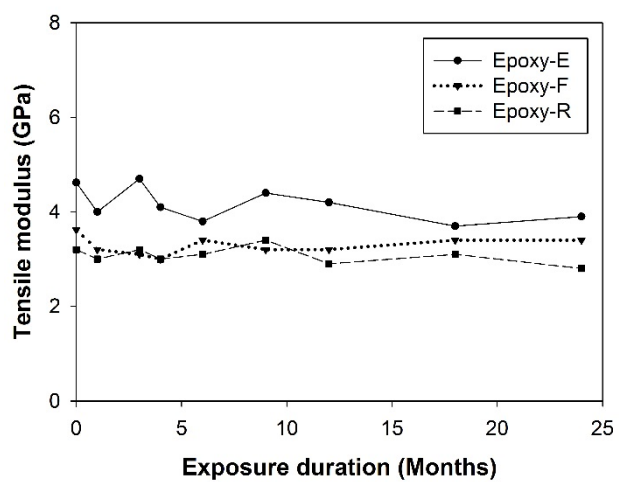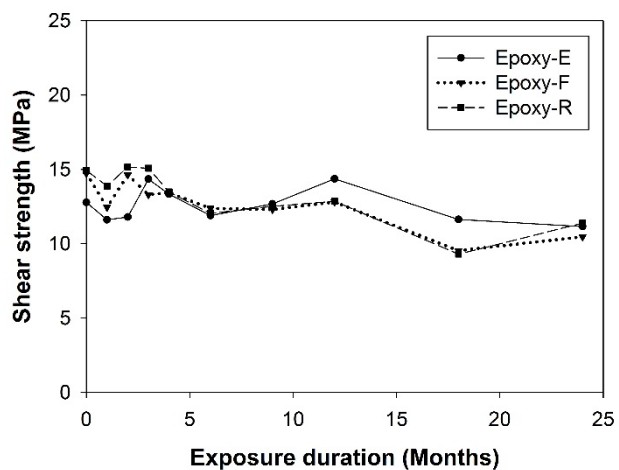
Fig. 5 Arrangement for the bond test

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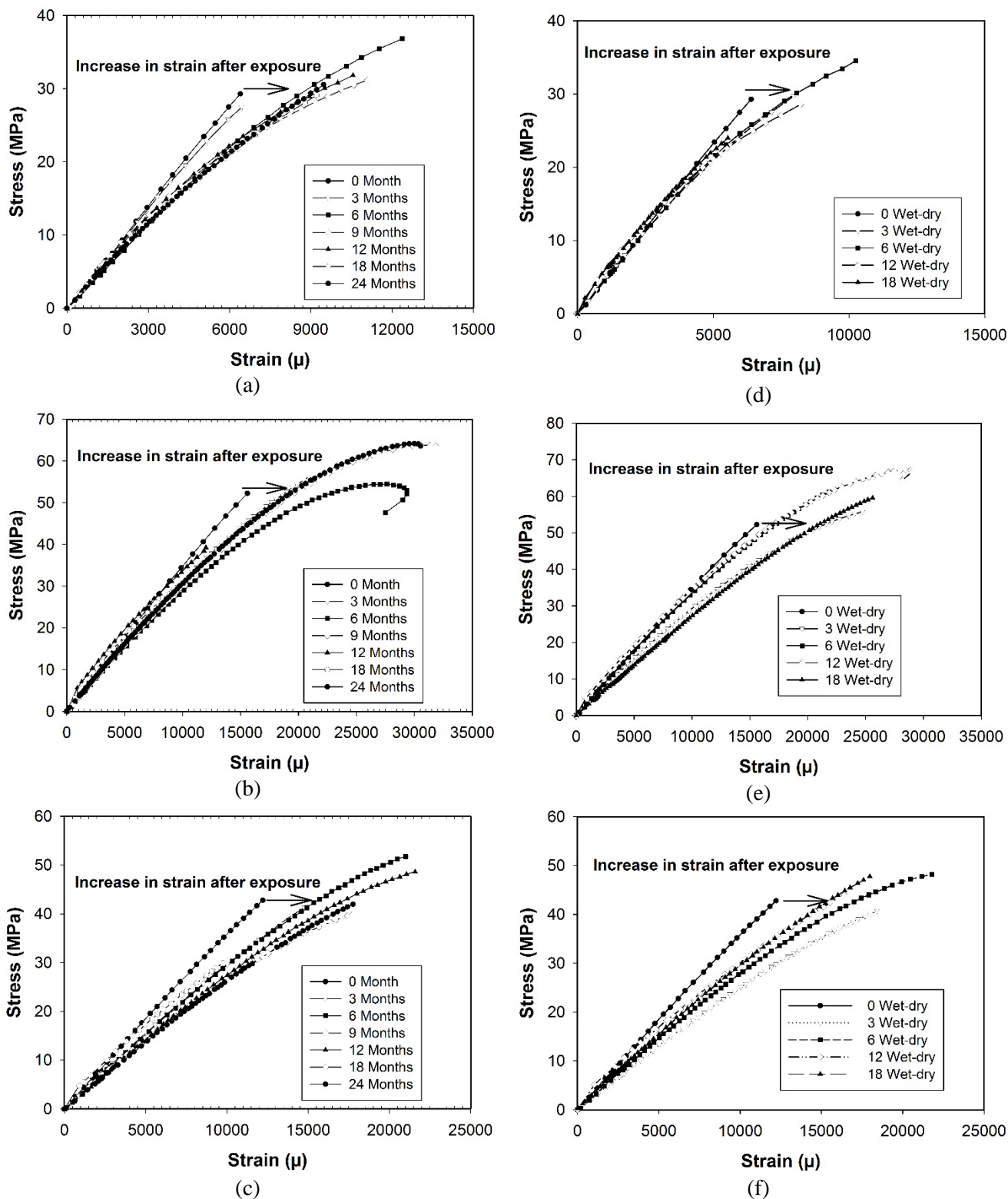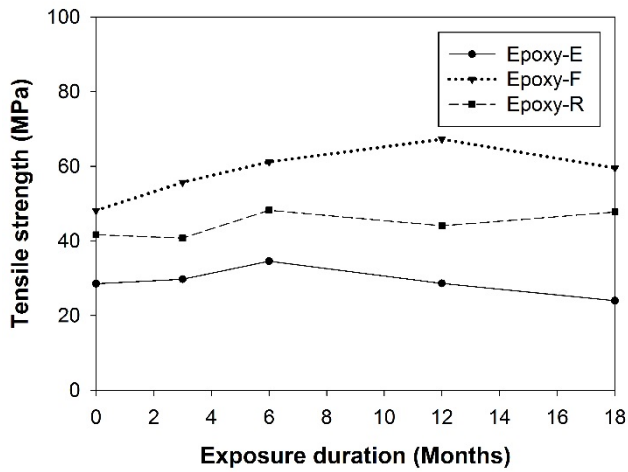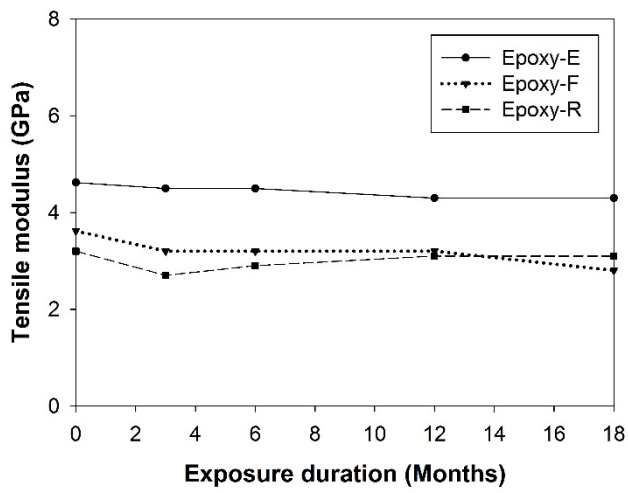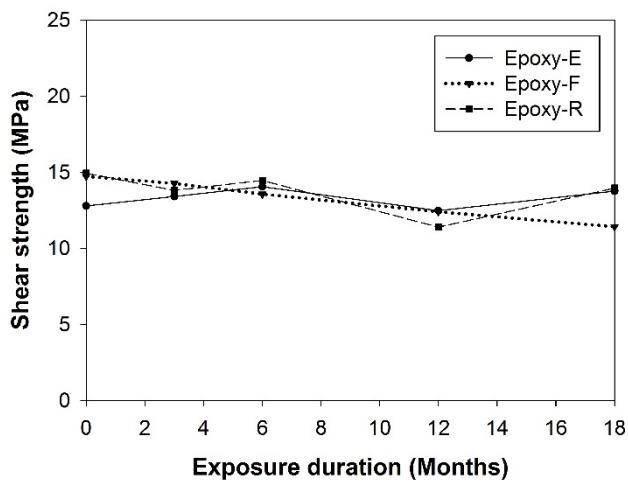
Fig. 8 Stress-strain curves for resins at different exposures: (a) Epoxy-E (Continuous immersion); (b) Epoxy-F (Continuous immersion); (c) Epoxy-R (Continuous immersion); (d) Epoxy-E (Wet-dry cycles); (e) Epoxy-F (Wet-dry cycles); and (f) Epoxy-R (Wet-dry cycles)

(a)

(b)

(c)

Fig. 9 Mechanical properties of the resin after wet-dry cycles: (a) Tensile strength; (b) Tensile modulus; and (c) Shear strength
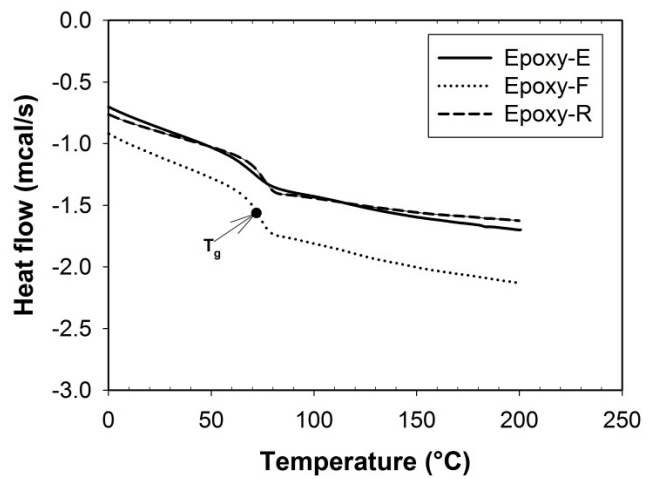
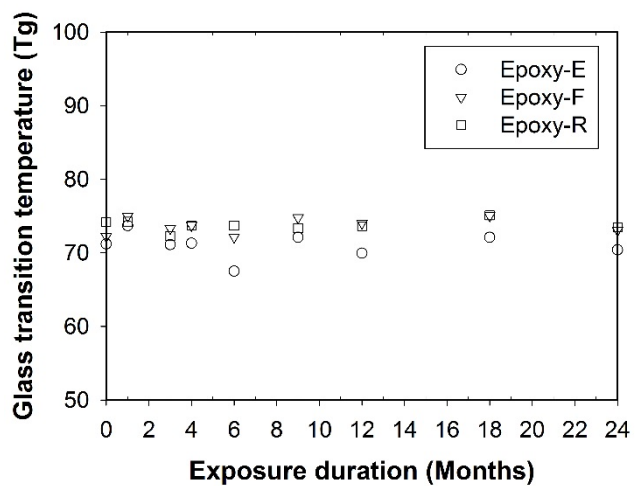**Fig. 10** Heat flow curves of epoxy resins before exposure

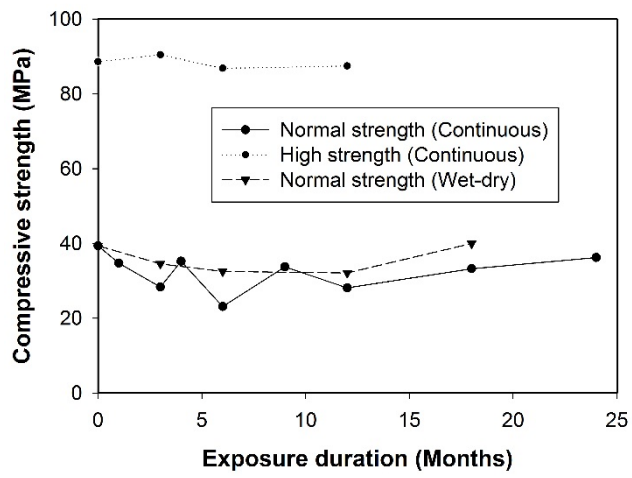**Fig. 11** Effect of continuous exposure on *Tg* of the resins after drying for a day
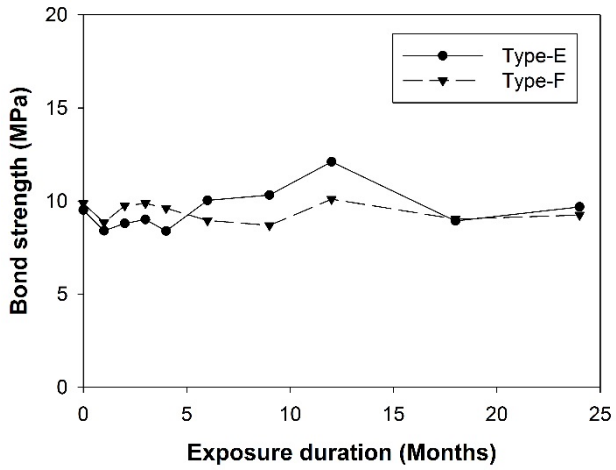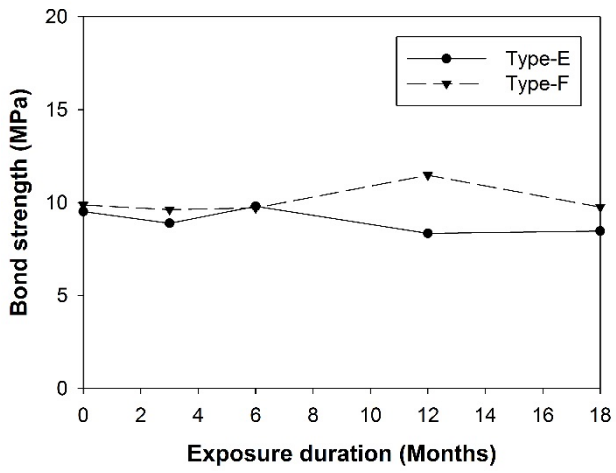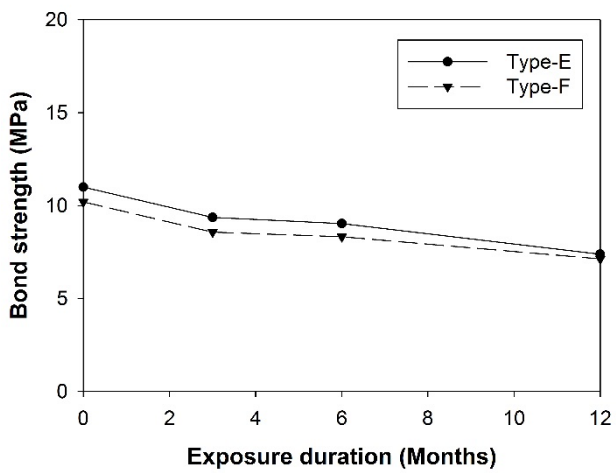
Fig. 12 Effects of exposure on concrete compression strength

(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
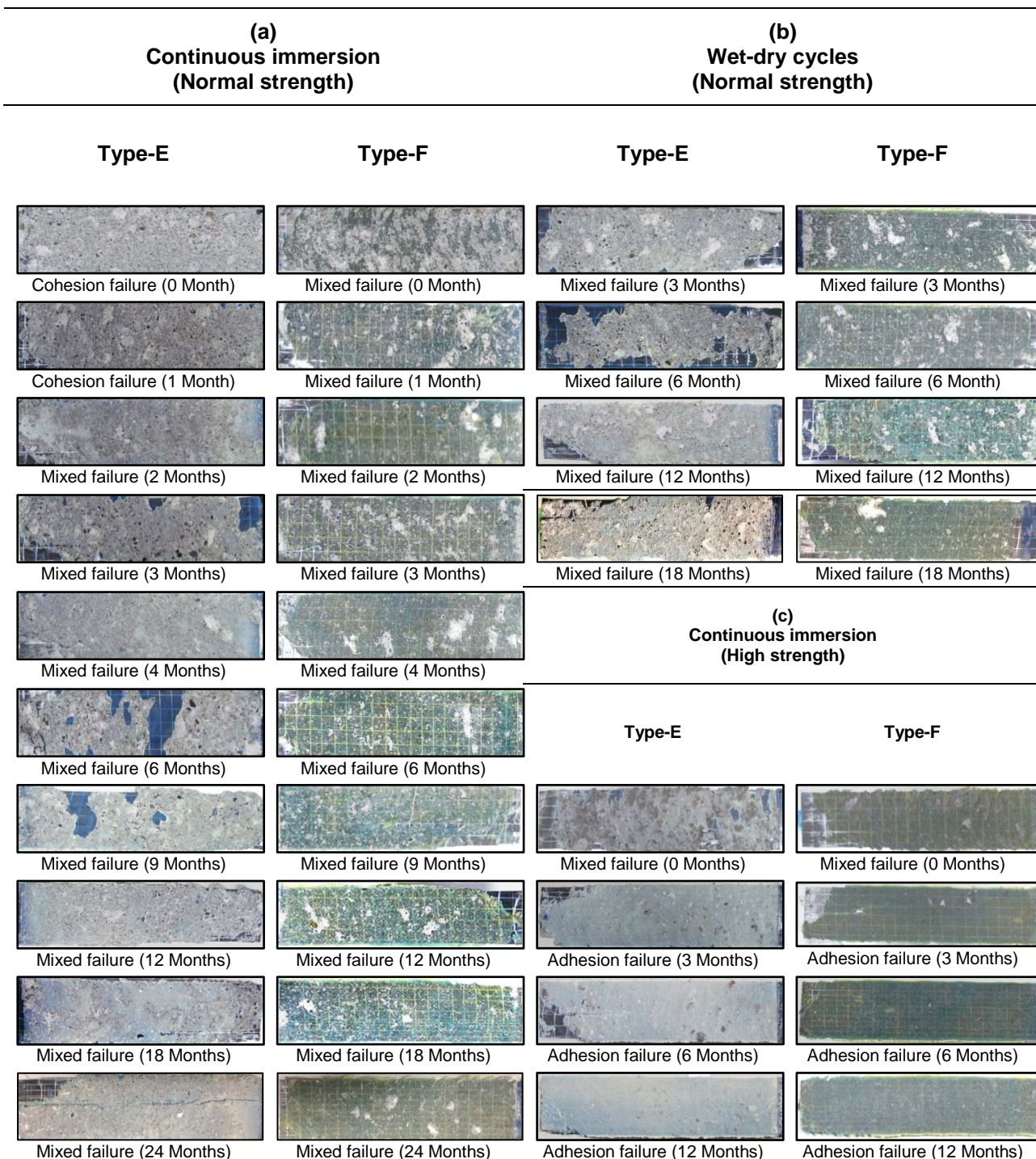
(a) **Continuous immersion** **(Normal strength)**		**(b)** **Wet-dry cycles** **(Normal strength)**	
Type-E	**Type-F**	**Type-E**	**Type-F**

(a) Continuous immersion (Normal strength)

Type-E | **Type-F**

- Cohesion failure (0 Month) | Mixed failure (0 Month)
- Cohesion failure (1 Month) | Mixed failure (1 Month)
- Mixed failure (2 Months) | Mixed failure (2 Months)
- Mixed failure (3 Months) | Mixed failure (3 Months)
- Mixed failure (4 Months) | Mixed failure (4 Months)
- Mixed failure (6 Months) | Mixed failure (6 Months)
- Mixed failure (9 Months) | Mixed failure (9 Months)
- Mixed failure (12 Months) | Mixed failure (12 Months)
- Mixed failure (18 Months) | Mixed failure (18 Months)
- Mixed failure (24 Months) | Mixed failure (24 Months)

(b) Wet-dry cycles (Normal strength)

Type-E | **Type-F**

- Mixed failure (3 Months) | Mixed failure (3 Months)
- Mixed failure (6 Month) | Mixed failure (6 Month)
- Mixed failure (12 Months) | Mixed failure (12 Months)
- Mixed failure (18 Months) | Mixed failure (18 Months)

(c) Continuous immersion (High strength)

Type-E | **Type-F**

- Mixed failure (0 Months) | Mixed failure (0 Months)
- Adhesion failure (3 Months) | Adhesion failure (3 Months)
- Adhesion failure (6 Months) | Adhesion failure (6 Months)
- Adhesion failure (12 Months) | Adhesion failure (12 Months)

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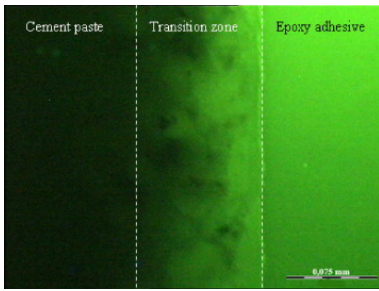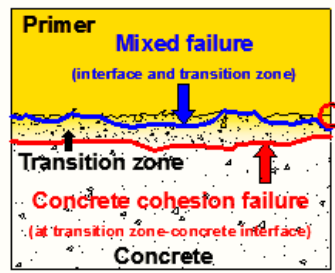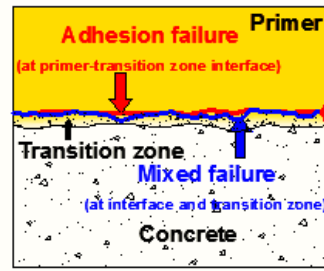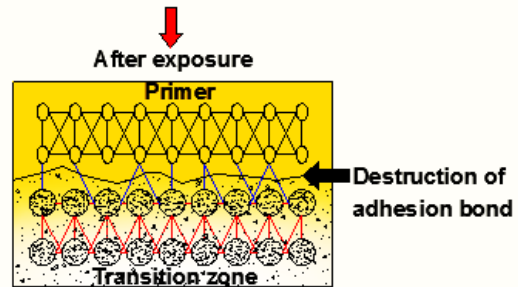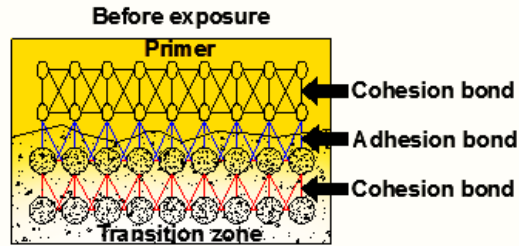
Fig. 15 Hardened cement paste-epoxy interface image obtained by optical microscopy under UV illumination (Djouani et al. 2011)

(a)   (b)

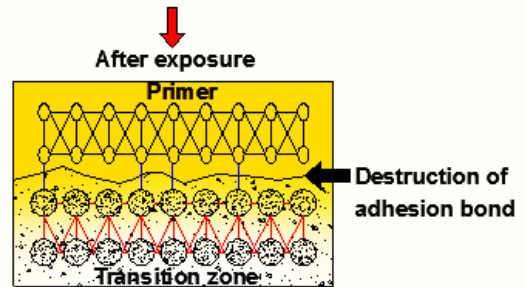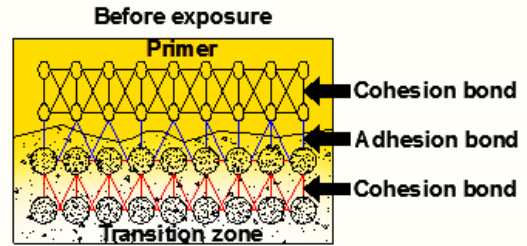**Fig. 16** Visualization of bond failure mechanism before and after exposure in (a) Normal strength concrete; and (b) High strength concrete
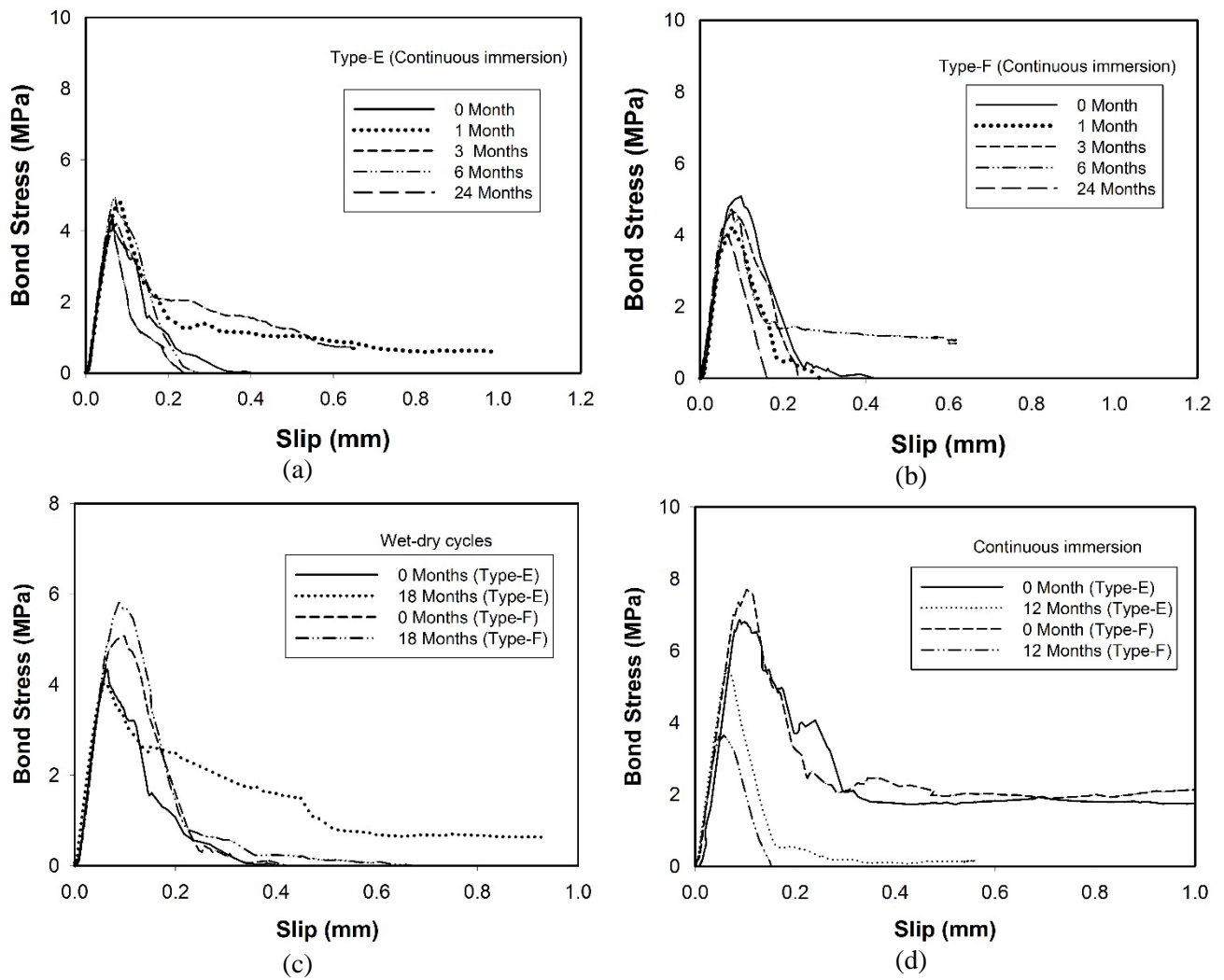
Fig. 17 Effect of moisture on local bond stress-slip curves at the same location for (a) normal strength concrete and continuous immersion (Type-E); (b) normal strength concrete and continuous immersion (Type-F); (c) normal strength concrete and wet-dry cycles; and (d) high strength concrete and continuous immersion
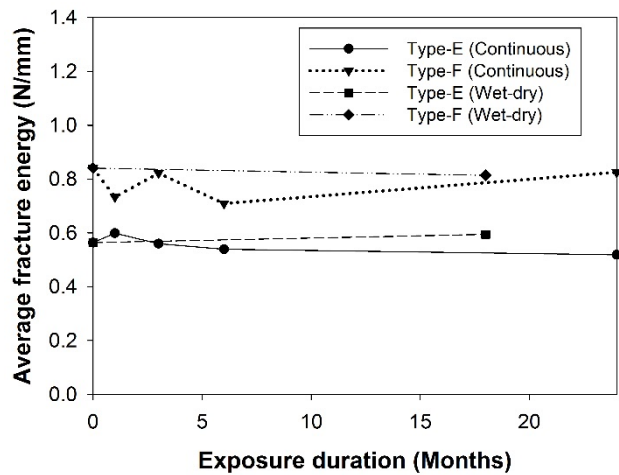
Fig. 18 Effect of moisture on average fracture energy for normal strength concrete

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

Authors	FRP system	Exposure conditions/duration	Test	Results	
				Bond strength	**Failure mode**
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
			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
	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
	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	3	-	-	-	-
1	1	1	1	1	-	1	-	-	-	-	-
2	1	1	1	1	-	1	-	-	-	-	-
3	1	1	1	1	1	1	1	1	1	1	1
4	1	1	1	1	-	1	-	-	-	-	-
6	1	1	1	1	1	1	1	1	1	1	1
9	1	1	1	1	-	1	-	-	-	-	-
12	1	1	1	1	1	1	1	1	1	1	1
18	1	1	1	1	-	1	-	1	1	1	1
24	1	1	1	1	-	1	-	-	-	-	-