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Effects of hygrothermal conditioning on epoxy adhesives used in FRP composites

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HIGHLIGHTS

- Six epoxies were tested to determine curing kinetics in hygrothermal environments.
- Competing effects of added cure and plasticization were observed in all epoxies.
- Significant decrease in T_g from theoretical values was observed in all clear epoxies.
- Average T_g of tested epoxies was below the design temperature for some U.S. bridge locations.

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ABSTRACT

Durability of FRP composites and their bond to concrete is essential to structural integrity of an FRP repair. Epoxy adhesives are used to form FRP composites, and as a bonding medium between the FRP and concrete substrate. Susceptibility of epoxy to the negative effects of water and high temperature affects the longevity of FRP repairs in hygrothermal environmental conditions. The presented work investigates the effects of such environments on the curing kinetics of epoxy. Competing effects of plasticization and post-cure during accelerated conditioning are discussed; recommendations targeting researchers, practitioners, and manufacturers are made based on the research findings.

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1. Introduction

FRP composites are deemed a prominent method for strengthening and repair of aged structures. However, their application in harsh environments is still inhibited by the poor resistance of FRP–concrete bond to such conditions. Due to the changing mechanical properties of adhesives (loss of stiffness) and their adhesion properties (loss of chemical bonds), when exposed to bond critical environments, the reliability of the FRP strengthening/repair scheme is compromised. Effects of environmental exposure, primarily moisture, on the integrity of epoxy–concrete bond is shown graphically in Fig. 1: in dry ambient conditions external load is completely transferred into the concrete substrate, allowing for distribution of damage in the substrate (Fig. 1a) at critical loading; however, when stiffness of epoxy and chemical bonding are adversely affected by the presence of moisture, full bond capacity cannot be attained, as the bond fails prematurely along the epoxy–concrete interface (Fig. 1b). Development of accelerated conditioning protocols (ACP) to assess the durability of FRP–concrete bonded systems is necessary in establishing more reliable design factors. Due to multiple degradation mechanisms existing in polymers when exposed to different environments, the selection of appropriate ACP is crucial in determining the durability of the bonded system.

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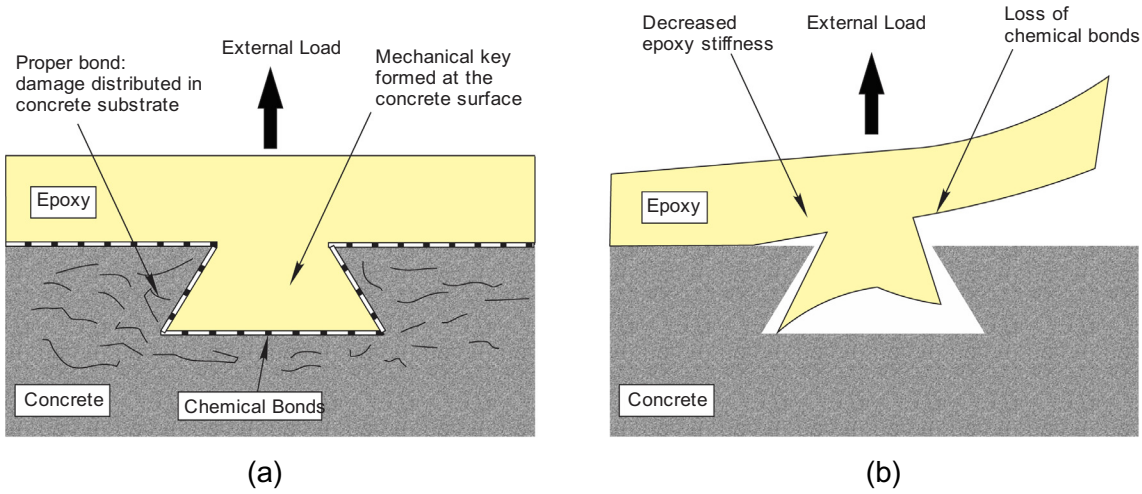

Fig. 1. Failure mode of epoxy–concrete bond: (a) in dry ambient conditions; (b) following exposure to moisture.

2. Motivation

Current industry testing standards for FRP composites used in civil infrastructure are prescribed by multiple internationally recognized technical organizations: AASHTO FRPS-1 [8], ACI 440.2M [10], and ICC AC125 [26]. To determine the durability properties of FRP, the composite is to be tested in direct tension following a prescribed ACP. ACP incorporate a range of exposure environments, among which some of them dictate a temperature of 60 °C, which can be slightly above the glass transition temperature of cold-cured epoxies used to form the composite.

In addition to testing the durability of FRP composites, multiple researchers ([1–3,21,33]) have concentrated their efforts on three-point bending beam test [24] to determine the durability properties of FRP–concrete bond. Three-point bending test method is currently a candidate for standardization, under the auspices of ASTM and ACI 440 subcommittee K – *FRP Material Characteristics*. The work of ACI 440 subcommittee L – *Durability* was focused primarily on specification of appropriate ACP to determine the durability of bond in three-point bending test. The main obstacle in this process was the lack of information in literature on determining the appropriate conditioning temperature for this purpose. While higher temperature is deemed to accelerate the degradation processes that take place at FRP–concrete bond, the main concern is that if the conditioning temperature is higher than the T_g, the mechanisms leading to loss of bond may be different than the mechanisms that occur during field exposure at lower temperatures. The purpose of this study is to provide a better understanding of evolution of degradation mechanisms in epoxy with time when exposed to hygrothermal ACP at temperatures lower than and higher than the measured T_g of epoxy adhesive.

3. Background

Structural epoxies used with FRP composites are available in two forms: *clear epoxy*, and more viscous *paste epoxy*. The difference in viscosity between the two types of adhesives comes from additives (such as silica particles) that are not found in clear epoxies. Epoxy adhesives used in construction are generally based on bisphenol A molecules hosting an epoxide functional group at both ends, forming the monomer in the epoxy structure [29]. Epoxy hardener is composed of amines that react with the epoxide groups to form covalent bonds. The amines bond the monomers together in a linear fashion to form polymer chains. They also allow for cross-linking between the polymer chains to form additional covalent bonds. The described process is termed "curing of epoxy"; with subsequent cross-linking a non-crystalline hardened molecular structure is formed [22]. At "full-cure" no additional cross-linking between the polymer chains is possible; at this point epoxy has reached its full mechanical properties. The cross-linking density is usually described in terms of conversion that takes values from 0 to 1.0, where a value of 1.0 signifies a fully-cured epoxy [16].

The bond between epoxy and concrete is established through chemical interactions and mechanical bonding [30]. While the exact nature of the chemical bond is not known, it is thought to consist of mostly hydrogen bonding between the surface molecules of concrete and epoxy [19]. Integrity of epoxy–concrete bond is considered to be primarily due to mechanical interlocking ([19,27]; Stewart, 2011; [30]). Stiffness of epoxy matrix, and consequently the integrity of its mechanical bond, can be compromised by two main degradation mechanisms: its transition to rubbery state at temperatures higher than the T_g, and plasticization of epoxy matrix.

Once the T_g is exceeded, the covalent bonds between the polymer chains are capable of rotating, but remain intact. Therefore, the general shape of the epoxy structure in glassy state is maintained, but its stiffness and strength are reduced. The value of T_g is dependent on polymer chain mobility. The lower the cross-linking density of the epoxy molecular structure, the less thermal energy is required for transition from a glassy state to a rubbery state [22,23]. Amounts of free volume and cross-linking (measured by conversion) affect polymer chain mobility and consequently change the epoxy T_g. At room temperature conditions for two or more weeks, the conversion of most commercially available structural epoxies ranges from 0.8 to 1.0, which means that 80–100% of possible covalent bonds are formed [34]. Exposure to temperatures above those experienced during initial curing will cause additional cross-linking [16]. Cross-linking restrains polymer chain mobility by interconnecting individual chains together; therefore, the higher the cross-linking density (more covalent bonds), the less chain mobility, and the higher T_g.

Chain mobility is also affected by the amount of free volume in the polymer structure. Free volume is the available space within the polymer chain network on a microscopic level [22,23]. With increase in free volume, the mobility of polymer chains increases as there is more available space for their movement; hence, less thermal energy is required to convert the epoxy into a rubbery state. Water absorption can lead to increases in free volume in

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