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Effects of Elevated Temperature Curing on Glass Transition Temperature of Steel/CFRP Joint and Pure Epoxy Adhesive

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ABSTRACT: Glass transition temperature (T_g) of the steel/epoxy/CFRP composite bond can affect the service and fire performance of the system. Two test series were conducted to evaluate the T_g of the pure epoxy adhesive and the steel/epoxy/CFRP bond. A total of twenty-six double strap joints and pure epoxy adhesive samples were prepared under different curing conditions to check the T_g of bond. Six different curing conditions were used. The test results revealed that the elevated temperature curing has a significant affect on the T_g of steel/epoxy/CFRP bond and pure epoxy adhesive. A considerable improvement of T_g was noted in the joint with elevated temperature curing when compare with the epoxy adhesive samples cured under the same condition. The strength degradation of the bond for a certain temperature exposure is also reduced with increased T_g of the joint.

KEYWORDS: Elevated temperature curing; Glass transition temperature; CFRP/steel joints; Epoxy adhesive; Service performance

1 INTRODUCTION

Glass transition temperature (T_g) is the temperature or temperature range where the polymer material changes its form to a soft rubbery state from a rigid glassy state (Becker & Locascio 2002). This change of its state causes for the rapid changes in mechanical properties of polymeric materials (Bai & Keller 2009, Bai et. al. 2008).

The CFRP strengthened steel structures are sensitive to the environmental changes due to the low T_g of polymeric adhesive. Due to low glass transition temperature of epoxy adhesive which is used to adhere CFRP to steel substrate, the degradation of mechanical properties may be expected even with the exposure to the daily cyclic temperatures. Nguyen et. al. (2011) noted a heavy strength degradation of steel/epoxy/CFRP joint with the exposure to temperatures near the T_g. A similar mechanical degradation at temperatures near T_g of epoxy adhesive has observed in concrete/epoxy/CFRP bond with the elevated temperature exposure (Gamage et. al.2016).

Mobility of the polymer chain of the epoxy increases when the epoxy temperature reaches its T_g (Petrie, 2006). This increment of mobility reduces the rigidity of the polymer chain, which will ultimately lead to a reduction of mechanical properties of the adhesive joint. However, if the rigidity of the polymer chain can be increased, T_g of the epoxy can also be increased (Wang et. al. 2011). This can be led to an increase the stability of bond.

Elevated temperature curing can be used to increase the T_g of bond result in reduced mechanical degradation of the bond (Nguyen et. al. 2013, Gamage et. al.2006). The increment of thermal properties with elevated curing temperature may lead to decrease the thickness of insulation to be applied on the composite to ensure required fire endurance (Ranasinghe et. al. 2011). The long term service performance of the bond over cyclic and humid environmental conditions can be increased with the elevated temperature curing (Gamage et. al.2016).

However, the curing methods applied in the control environment are not practical enough to use in large Civil Engineering applications. Therefore, this study focus on a practical curing method, which can be applied for the Civil Engineering applications. The performance of the bond due to new curing method will be compared with the T_g of pure epoxy adhesive

2 TEST PROGRAMME

Two series of test programmes were conducted to find the glass transition temperature (Tg) of the steel/epoxy/CFRP joints and pure epoxy adhesive. Effects of elevated temperature curing in Tg were studied. Effects of six different elevated temperature curing conditions were examined.

Table 1. Measured and manufacturer provided material properties

	Material	Tensile strength (MPa)	Ultimate strain	Elastic modulus (GPa)	Poison's ratio
Measured	Steel (ASTM A 370-02)	583	0.065	200	0.3
	Adhesive (ASTM D 638-a)	25	0.043	0.977	0.3
	CFRP (ASTM D 3039)	1575	0.009	175.62	0.3
Manufacturer Provided	Adhesive (ARELDITE 420 A/B) *	29	0.056	1.495	0.3
	CFRP (X-Wrap C300)	4000	0.02	240	0.3

*T_g of adhesive- 55 ^{0}C

2.1 *Material properties*

Measured and manufacturer provided material properties are shown in Table 1. An average 60% and 55% discrepancies were observed in measured and manufacturer provided material properties for CFRP materials in tensile strength and ultimate strain, respectively. Manufacturer provided T_g was 55 ^{0}C for ambient curing conditions.

2.2 *Sample preparation and testing*

2.2.1 *CFRP/epoxy/steel bond*

The wet lay-up method was used in the fabrication of CFRP/steel double strap joints (Fawzia et. al. 2006). Two steel plates with 190 mm length, 40 mm width and 4 mm thickness were used for the double strap joints (Figure 1). Bond length of 140 mm was selected as the effective bond length for the samples with the same condition is 120 mm (Chandrathilaka et. al. 2018). A K-type thermo-couple was fixed in the interface between steel and epoxy to measure the temperature in the bond line through curing stage and during testing. Thermo-couples were properly calibrated before fixing (Figure 1 (b)).

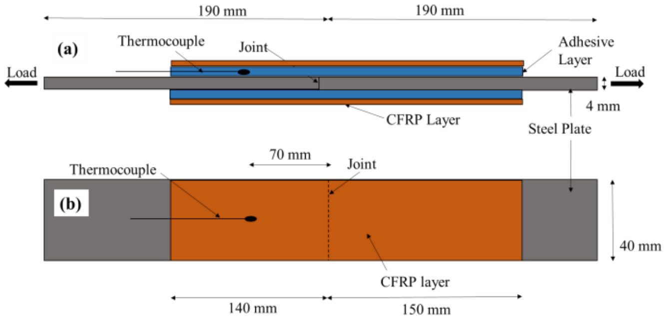

Figure 1. Schematic diagram of double strap joint, (a) cross section, (b) plan view

Total number of twenty-six double strap joints (Figure 2) were prepared. Twelve of them (control samples) were cured at ambient temperature (30 ^{0}C) for 7 days. Other fourteen samples were initially cured at average elevated temperature of 75 ^{0}C for four hours before curing under ambient temperature for 7 days. The initial elevated temperature cured samples were allowed a ±5 ^{0}C margin of temperature variance due to non-controllable practical nature. A set of halogen floodlights with the 1000 W capacity were used to cure the samples in elevated temperature [Figure 3].

Figure 2. Prepared sample

Figure 3. Elevated temperature curing using halogen floodlights

The ultimate strength of the double strap joints was determined using the Universal testing machine with 1000 kN capacity as shown in Figure 4. The environmental temperature was raised to reach the bond line temperature to 30 ^{0}C, 50 ^{0}C, 60 ^{0}C, 70 ^{0}C, 80 ^{0}C, 90 ^{0}C and 100 ^{0}C before testing. Two identical samples were tested under each condition. Before the testing of a certain sample at its testing temperature, it was allowed to stabilize its bond line temperature to the testing temperature. An average time duration of 10 minutes was assigned to stabilize its bond line temperature.

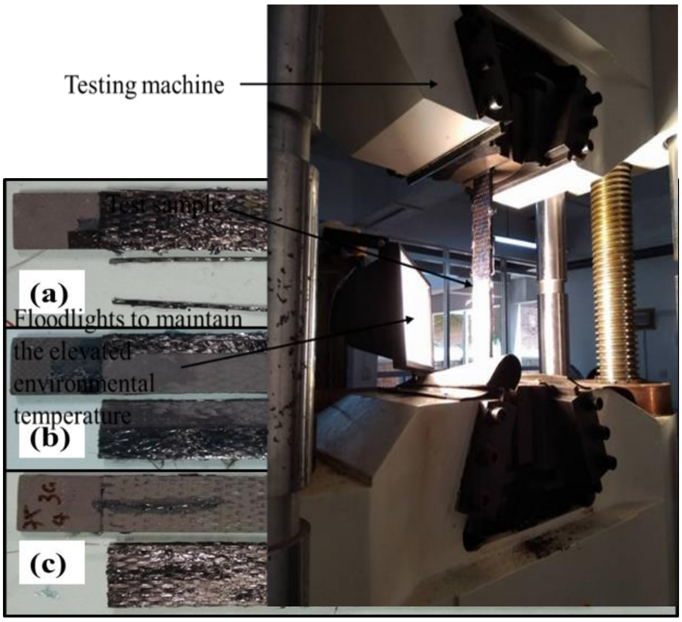

Figure 4. Testing apparatus

2.2.2 *Pure epoxy adhesive*

Six samples of epoxy adhesive were cured under six different curing conditions as shown in Table 2. “CA” sample was cured at ambient temperature as a control sample with providing the same curing conditions as the control double strap joint samples. “EO” sample was initially cured at 75 ^{0}C for one hour using a standard oven. Samples EF1, EF2, EF3 and EF4 were initially cured using the floodlight system described in Figure 3. After the initial elevated temperature curing all prepared samples were kept to cure for 7 days under ambient temperature condition.

The pure epoxy samples were tested using a Differential Scanning Colorimeter (DSC). A heat rate of 2 ^{0}C/min was applied during the testing. Alumina T zero pan with a T zero hermetic lid was used to hold the sample while heating.

Table 2. Initial curing configuration of epoxy adhesive

Sample	Elevated curing temp. (^{0}C)	Curing time (hours)	Curing method
CA	Ambient	N/A	Ambient
EO	75	1	Oven
EF1	75±5	1	Floodlights
EF2	55±5	1	Floodlights
EF3	75±5	2	Floodlights
EF4	75±5	4	Floodlights

3 TEST RESULTS

3.1 *Failure Loads and failure modes of double strap joints*

Average failure loads and failure mechanisms of each specimen type were listed in Table 3. The results indicate a trend of decreasing the average failure load with the bond line temperature for both curing conditions. However, the elevated temperature cured samples have shown higher failure loads than the ambient temperature cured samples at similar bond line temperatures. In the range of 7% to 78% difference in failure loads were noted between ambient and elevated temperature cured samples when the bond line temperature is below 50 ^{0}C. When the bond line reaches the temperature range of 60 ^{0}C and 80 ^{0}C, the elevated temperature cured samples indicated a relatively higher strength. With the bond line exceeds 90 ^{0}C, this difference was negligible.

The observed failure mechanisms are shown in Figure 5. CFRP fiber rupture was observed only in the samples tested at 30 ^{0}C. When the bond line temperature increases, the failure mode was shifted from CFRP fiber rupture to adhesive-steel interface debonding. Ambient temperature cured samples had shown mix failure mode when the interface temperature reaches the range from 50 ^{0}C to 80 ^{0}C. However, the elevated temperature cured samples were failed due to an adhesive steel interface debonding when the bond line temperature reached 60 ^{0}C.

Figure 5. Major failure modes of double strap joints, (a) CFRP fiber rupture, (b) CFRP rupture and interface debonding, (c) Adhesive-steel interface debonding

3.2 *Glass transition temperature of double strap joints (T_g)*

The glass transition temperature was calculated as shown in Figure 6. T_g for ambient temperature cured sample and elevated temperature cured sample were 50 ^{0}C and 61 ^{0}C, respectively. On average, 22% increment in T_g of bond can be seen with the elevated temperature curing. Decreasing of the load with the bond line temperature for both curing conditions have shown a similar pattern, which is common for the polymeric adhesives. However, the strength reduction of the ambient temperature cured samples were initiated at 40 ^{0}C which was started after 50 ^{0}C for elevated temperature cured samples. Similar behavior was noted when the bond line reaches the temperatures of 90 ^{0}C and 100 ^{0}C.

Table 3. Failure loads and mechanisms

Curing condition	Bond line temperature at testing (^{0}C)	Average failure load (kN)	Failure mode
Ambient temperature curing	30	36.83	CFRP fiber rupture
	50	33.4	CFRP rupture and interface debonding
	60	26.05	CFRP rupture and interface debonding
	70	21.18	CFRP rupture and interface debonding
	80	15.5	CFRP rupture and interface debonding
	90	15.63	Adhesive-steel interface debonding
Elevated temperature curing	30	40.05	CFRP fiber rupture
	50	37.8	CFRP rupture and interface debonding
	60	33.6	Adhesive-steel interface debonding
	70	31.13	Adhesive-steel interface debonding
	80	27.6	Adhesive-steel interface debonding
	90	16.7	Adhesive-steel interface debonding
	100	15.05	Adhesive-steel interface debonding

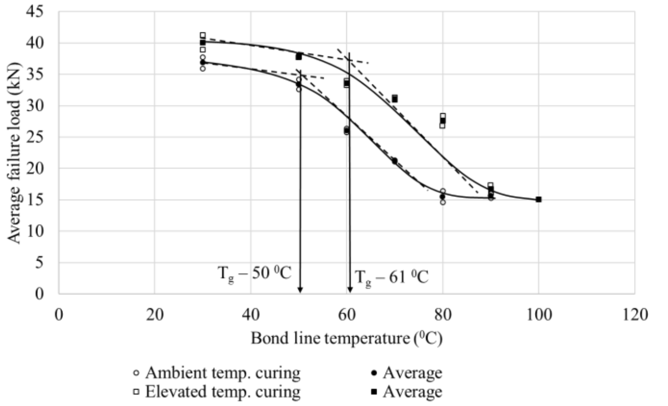

Figure 6. Glass transition temperature of CFRP/epoxy/steel joint

3.3 *Glass transition temperature of pure epoxy adhesive*

Differential scanning calorimetry (DSC) was used to measure the T_g of pure epoxy adhesive. Heat flow with the temperature has shown in Figure 7 for tested samples. The T_g values are listed in Table 4. The T_g of epoxy had increased by 19% with elevated temperature curing at 75 ^{0}C for 4 hours. When the elevated temperature curing at 55 ^{0}C was done, the T_g has not affected considerably. The oven cured sample had shown a 3% reduction in T_g with samples cured using the floodlights at the same curing temperature and period. Curing period had a significant effect on T_g as it can increase the T_g up to 4% with increasing the curing time from one hour to four hours.

Table 4. T_g of pure epoxy adhesive

Sample	T_g (^{0}C)
CA	49.2
EO	54.1
EF1	55.9
EF2	49.1
EF3	57.0
EF4	58.2

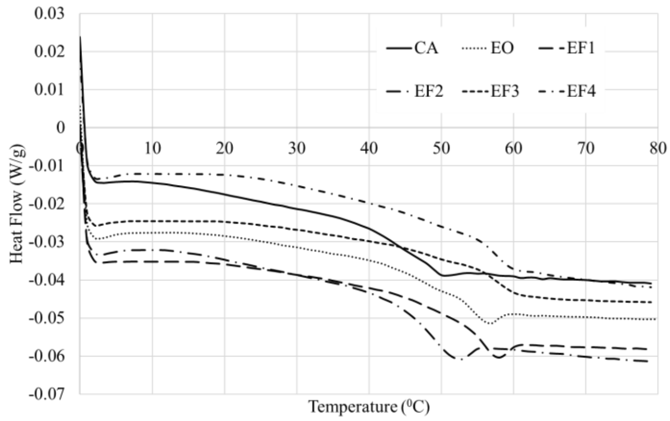

Figure 7. Heat flow vs temperature for pure epoxy adhesive

4 COMPARISON BETWEEN PURE EPOXY BEHAVIOR AND BOND BEHAVIOR

For the specimens cured at ambient conditions the T_g were almost similar for the epoxy adhesive and CFRP/epoxy/steel joints. A slightly lower value (1.6%) was observed from the pure epoxy adhesive. The pure epoxy sample had shown a 2.8 ^{0}C lower value of T_g, compared to the steel/epoxy/CFRP double strap joints with elevated temperature curing. The increase of T_g for the bond may happen due to the composite action of the double strap joints with elevated temperature curing. T_g of CFRP material is very high compared to the pure epoxy adhesive. Therefore, the composite action of double strap joints might cause for increasing of T_g of bond by a small amount compared to the pure epoxy adhesive.

5 CONCLUSIONS

Two test series were conducted to determine the T_g of pure epoxy adhesive and the steel/epoxy/CFRP double strap joints. Two curing conditions were used in the preparation of steel/epoxy/CFRP double strap joints, while six curing conditions were used in the pure epoxy adhesive samples. The degradation of mechanical properties of bond and T_g of pure epoxy adhesive were examined after exposure to elevated temperature. The following conclusions were made;

a. The ambient temperature cured samples have shown the initiation of rapid strength reduction at 40 ^{0}C while the elevated temperature cured samples had shown the same behavior at 50 ^{0}C. The same trend of strength reduction of CFRP/steel joint was noted with the exposure to elevated temperature, irrespective of the curing conditions.
b. Failure mode has shifted from CFRP fiber rupture to adhesive steel interface debonding in double strap joints with the increased bond line temperature, for both curing conditions. CFRP fiber rupture has seen only from the specimens tested at 30 ^{0}C bond line temperature. This shows evidence for the strength degradation of bond with exposure to the elevated temperature.
c. Elevated temperature curing has increased the T_g of bond from 50 ^{0}C to 61 ^{0}C with increasing the curing temperature from 30 ^{0}C to 75 ^{0}C.
d. T_g of pure epoxy adhesive is proportional to the curing temperature and curing period of the samples. However, 55 ^{0}C cured samples did not show a significant increase of T_g compared to the 75 ^{0}C cured samples.
e. T_g of steel/epoxy/CFRP bond is slightly higher than the T_g of pure epoxy bond. On average, 1.6% and 4.7% increments were observed in the ambient temperature cured (control) and elevated temperature cured samples (at 75 ^{0}C for four hours), respectively.
f. The manufacturer provided T_g for epoxy adhesive was 55 ^{0}C. This is 10% greater than the measured T_g of epoxy adhesive under ambient condition. T_g of pure epoxy adhesive was almost similar to T_g of joint cured under the ambient condition. Use of T_g provided by the manufacturer for epoxy adhesive in designing for fire or service performances of the CFRP/steel composite is problematic.

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7 REFERENCES

Becker, H., and Locascio, L.E., “Polymer microfluidic devices”, Talanta, Vol. 56, 2002, pp 267–87.

Bai, Y., and Keller, T., “Modeling of mechanical response of FRP composites in fire”, Composites: Part A, Vol. 40(6-7), 2009, pp 731–8.

Bai, Y., Keller, T., and Vallee, T., “Modeling of stiffness of FRP composites under elevated and high temperatures”, Compos Sci Technol, Vol. 68(15-16), 2008, pp 3099–106.

ARELDITE 420 A/B, “Two component epoxy adhesive system”, Huntsman Advanced Materials, 2009.

Nguyen, T., Bai, Y., Zhao, X., and Al-mahaidi, R., “Mechanical characterization of steel / CFRP double strap joints at elevated temperatures”, ,Compos Struct, Vol. 93, 2011, pp 1604–1612.

Gamage, J.C.P.H., Al-mahaidi, R., and Wong, M.B., “Integrity of CFRP-concrete bond subjected to longterm cyclic temperature and mechanical stress”, Compos Struct, Vol. 149, 2016, pp 23–33.

Petrie, E.M., “Handbook of Adhesives and Sealants”, McGraw-Hill Handbooks, 2006.

Wang, R., Zheng, S., and Zheng, Y., “Polymer matrix composites and technology”, Woodhead Publishing Limited and Science Press Limited, 2011, pp.101-167.

Nguyen, T., Bai, Y., Zhao, X., and Al-mahaidi, R., “Curing effects on steel / CFRP double strap joints under combined mechanical load, temperature and humidity”, Constr Build Mater, Vol. 40, 2013, pp 899– 907.

Gamage, J.C.P.H., Al-Mahaidi, R., and Wong, M.B., “Bond characteristics of CFRP plated concrete members under elevated temperatures”, Vol. 75, 2006, pp. 199–205.

Ranasinghe, R.A.T.M., Jinadasa, D.V.L.R., Srilal, H.P.S., and Gamage, J.C.P.H., “Bond performance of cfrp strengthened concrete subjected to fire”, Civil Engineering Research for Industry, Sri Lanka, 2011, pp 37-42.

ASTM A 370-02, “Standard Test Methods and Definitions for Mechanical Testing of Steel Products”, West Conshohocken, PA, USA: ASTM International. 2003.

ASTM D 638 - 2002a, "Standard Test Method for Tensile Properties of Plastics", West Conshohocken, PA, USA: ASTM International. 2002.

ASTM D 3039/3039M – 00, "Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials", West Conshohocken, PA, USA: ASTM International. 2000.

X-Wrap C300, "High strength carbon fiber fabric for structural strengthening", X-CALIBUR structural systems,

Fawzia, S., Al-mahaidi, R., and Zhao, X., "Experimental and finite element analysis of a double strap joint between steel plates and normal modulus CFRP" Composite Structures, Vol. 75, 2006; pp 156–162.

Chandrathilaka, E.R.K., Pererea, U.N.D., and Gamage, J.C.P.H., "Bond slip models for corroded steel-CFRP double strap joints", 6th international symposium of advances in Civil and Envoirenmental Engineering Practices for sustainable development, Sri Lanka, 2018, pp 310-317.