

Article

The Feasibility of Modified Magnesia-Phosphate Cement as a Heat Resistant Adhesive for Strengthening Concrete with Carbon Sheets

Ailian Zhang ^{1,2} and Xiaojian Gao ^{1,*}¹ School of Civil Engineering, Harbin Institute of Technology, Harbin 150001, China; 12B333003@hit.edu.cn² School of Civil Engineering, Sichuan College of Architectural Technology, Deyang 618000, China

* Correspondence: gaoxj@hit.edu.cn; Tel.: +86-451-8628-1118

Academic Editor: Andrea Paglietti

Received: 6 May 2016; Accepted: 14 June 2016; Published: 17 June 2016

Abstract: External bonding of carbon fiber sheets has become a popular technique for strengthening concrete structures all over the world. Epoxy adhesive, which is used to bond the carbon fiber sheets and concrete, deteriorates rapidly when being exposed to high temperatures. This paper presents a high-temperature-resistant modified magnesia-phosphate cement (MPC) with the compressive strength that does not decrease at the temperature of 600 °C. The bond properties of both the modified MPC and the epoxy adhesive between externally bonded carbon fiber sheets and concrete were evaluated by using a double-shear test method after exposure to elevating temperatures from 105 °C to 500 °C. The results showed that the bond strength of the modified MPC at room temperature (RT) is much higher than that of the epoxy resin. Full carbonation with almost 0 MPa was detected for the epoxy sample after the exposure to 300 °C, while only 40% reduction of bond strength was tested for the modified MPC sample. Although the modified MPC specimens failed through interlaminar slip of fiber strips instead of complete debonding, the MPC specimens performed higher bond strength than epoxy resin at ambient temperature, and retained much higher bond strength at elevated temperatures. It could be concluded that it is feasible to strengthen concrete structural members with externally bonded carbon fiber sheets using the modified MPC instead of epoxy adhesive. Furthermore, the use of the modified MPC as the binder between carbon fiber sheets and concrete can be less expensive and an ecologically friendly alternative.

Keywords: strengthening concrete; carbon fiber sheets; epoxy resin; modified magnesia-phosphate cement (MPC); resistance to elevated temperatures

1. Introduction

Carbon fiber reinforced polymer (CFRP) has become in recent years one of the state-of-the-art materials in repairing and/or strengthening structural concrete elements since it has numerous advantages such as extremely high strength to weight ratio, versatility, and resistance to electrochemical corrosion [1]. However, CFRP materials are too sensitive to elevated temperatures [2]. Deterioration in mechanical and/or bond properties can be expected at temperatures approaching the glass transition temperature (T_g) of the organic epoxy adhesive [2], which is typically lower than 100 °C [3–5]. The replacement of organic epoxy adhesive with some inorganic adhesives is a promising solution. The inorganic adhesives such as magnesium oxychloride cement (MOC) and alkali activated cementitious materials [6] were used for organic epoxy adhesives replacement due to the favorable binding properties, non-toxic, low cost and excellent heat-resistance.

Several studies have been performed on the use of alkali activated cementitious materials as a sole binding material to expand the applied range of CFRP [7–9]. Kurtz et al. [7] reported

that the alkali activated cementitious material increases the strength and stiffness of reinforced concrete beams as effectively as organic epoxy, with a minor reduction in ductility. Toutanji et al. [8] presented that the increase of fatigue strength of concrete beam due to the use of alkali activated cementitious materials pasting carbon fiber sheets reaches as high as 55% when being compared with unstrengthened beams. Katakalos et al. [9] reported that reinforced concrete beams strengthened by alkali activated cementitious materials have good fatigue performance. On the other hand, alkali activated cementitious material shows a significant shrinkage, as many studies reported [10], which could easily generate cracking between carbon fiber sheets and concrete specimen. MOC cement has many properties superior to the conventional Portland cements, including higher compressive and tensile strength, better fire resistance, lower shrinkage and creep and better durability, but the application has been limited by its poor water resistance, warping deformation, efflorescence, cracking and reinforced corrode by chloride ion [11,12].

Magnesia-phosphate cement (MPC) is based on the chemical reaction of acidic liquid or phosphate with a solid basic dead-burned magnesia oxide powder [13]. This cement has several advantages such as high early strength, rapid setting and hardening, excellent bonding with old Portland cement concrete [14–17]. The use of this cement as a rapid patch repair material in construction field has been well studied in the past decades [14,18,19]. However, this material has seldom been used as an adhesive for concrete strengthening, especially in high temperature environments.

This paper seeks to investigate the bond properties of the modified MPC between externally bonded carbon fiber sheets and concrete at elevated temperatures when being compared with epoxy resin. The compressive strength of the modified MPC does not decrease at the temperature of 600 °C [20]. The specimens bonded by different adhesives (epoxy resin and the modified MPC) were heated to different temperature levels of 105 °C, 200 °C, 300 °C, 400 °C and 500 °C and maintained for 3 h. The bond strength and failure types of specimens after different high temperature exposures were measured.

2. Experimental

2.1. Raw Materials

The modified MPC was prepared by blending dead-burned magnesia powder (MgO), ammonium dihydrogen orthophosphate (ADP, $\text{NH}_4\text{H}_2\text{PO}_4$), borax (B), wollastonite powder (CaSiO_3) and tap water together. The first three ingredients were industrial-grade of purity 95%–99%. The wollastonite consists of 45.6% of CaO and 48% of SiO_2 , mainly in amorphous form and has an average specific surface area of 842.7 m^2/kg . Based on the former study (unpublished) [20], three binder proportions were designed as presented in Table 1. The molar ratio of Wollastonite and MgO to ADP was kept at 9:1. MA1 is the control mix for MPC. Based on the control mix, wollastonite was added to replace 5% and 10% (in molar percent) of MgO dosage. The mass ratio of water to powder was kept at 0.15. The borax was added by 5% of MgO weight to control setting time. The organic adhesive used in this paper was a two-part epoxy resin. The epoxy resin consists of parts A and B that are blended at a weight ratio of 8:2, and stirred until a homogeneous mixture is obtained. Unidirectional carbon fiber sheets were used. The mechanical properties of the carbon fiber sheets and epoxy resin provided by the manufacture are shown in Table 2.

Table 1. Details of the designed binder proportions.

Mix	Wollastonite (Molar Percent)	MgO (Molar Percent)	Weight Dosage		
			Wollastonite	MgO	ADP
MA1 (control)	0	100	0	100	31.9
MA2 (5%)	5	95	15.3	100	33.6
MA3 (10%)	10	90	32.2	100	35.5

MgO is dead-burned magnesia powder and ADP is ammonium dihydrogen orthophosphate.

Table 2. Mechanical properties of carbon fiber sheets and epoxy resin.

Property	Carbon Fiber Sheets	Epoxy Resin
Elastic Modulus (GPa)	244	19
Tensile strength (MPa)	4125	39.12
Areal Density (g/m ²)	300	-
Nominal Thickness (mm)	0.167	-

For interfacial bond testing, concrete with dimensions of 100 mm × 100 mm × 100 mm was prepared in the laboratory, respectively, with the following three mixing proportions: cement (319, 370 and 425 kg/m³), water (179, 170 and 170 kg/m³), sand (755, 784 and 801 kg/m³), coarse aggregate (1042, 1043 and 1051 kg/m³) and superplasticizer (5.42, 4.25 and 8.50 kg/m³). The concrete specimens were cured for 28 days in a fog room before the bond strength test. To protect the carbon fiber sheets from fire damage during the high temperature treatment, a tunnel fire coating was used which is produced by the Beijing Mercutio Fire Source Factory (China). This tunnel fire coating has a density of 600 kg/m³, a specific heat 1000 J/(kg·K) and a heat conductivity coefficient 0.12 W/(m·K).

2.2. Testing Methods

2.2.1. Bond Strength

The bond property between carbon fiber sheets and concrete is a key factor controlling the behaviour of the strengthened concrete elements. There are many different experimental methods used for determining the bond strength. Yao *et al.* [21] classified the existing measurement approaches into the following three types (see Figure 1): (a) single-shear test; (b) double-shear test; and a (c) beam test. Due to the simplicity and reliability [22], the double-shear test was adopted in this study for measuring the bond strength between the carbon fiber sheets and concrete specimen.

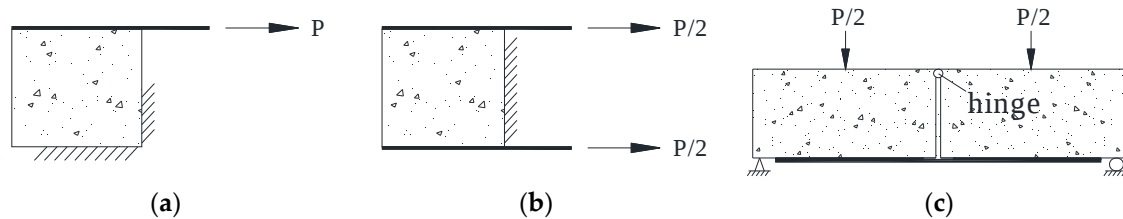


Figure 1. Sketch of test methods for interfacial bond properties: (a) single-shear test; (b) double-shear test; (c) beam test.

During the double-shear test, the loading was imposed on the prepared specimen by using a computer controlled electronic universal testing machine until failure, under a displacement control at a speed of 0.2 mm/min. The critical load at failure was recorded and the bond strength can be calculated according to the following equation:

$$\tau = \frac{P_u}{2b_f L_f} \quad (1)$$

In the above equation, τ = bond strength (MPa); P_u = critical load imposed on the specimen at failure (kN); b_f = the bonding width of carbon fiber sheets (mm); and L_f = the bonding length of carbon fiber sheets (mm).

2.2.2. Preparation of Specimens

For the double-shear test, the carbon fiber sheets were pasted on a pair of opposite surfaces for each concrete specimen and the procedure (as shown in Figure 2) was described as below.

(a) The concrete surface was cleaned using a brush to remove dust particles and then was wet by some water.

(b) A one-millimetre-thick primer layer of MPC or epoxy resin was trowelled to the concrete surface uniformly by using a spatula. Then, the specimen was kept to cure until MPC or epoxy resin started to lose flowability or become thicker.

(c) The carbon fiber sheets were pressed onto the above-preprocessed surfaces by using a ribbed roller ensuring the required bond width and length of 70 mm and 100 mm respectively. At the same time, air bubbles entrapped in the MPC or epoxy resin layer were rolled out by this treatment.

(d) To provide the same protection for carbon fiber sheets from fire damage during high temperature treatment, a 2-mm thick layer of MPC was coated on the outer surface of the carbon fiber sheets for all the specimens.

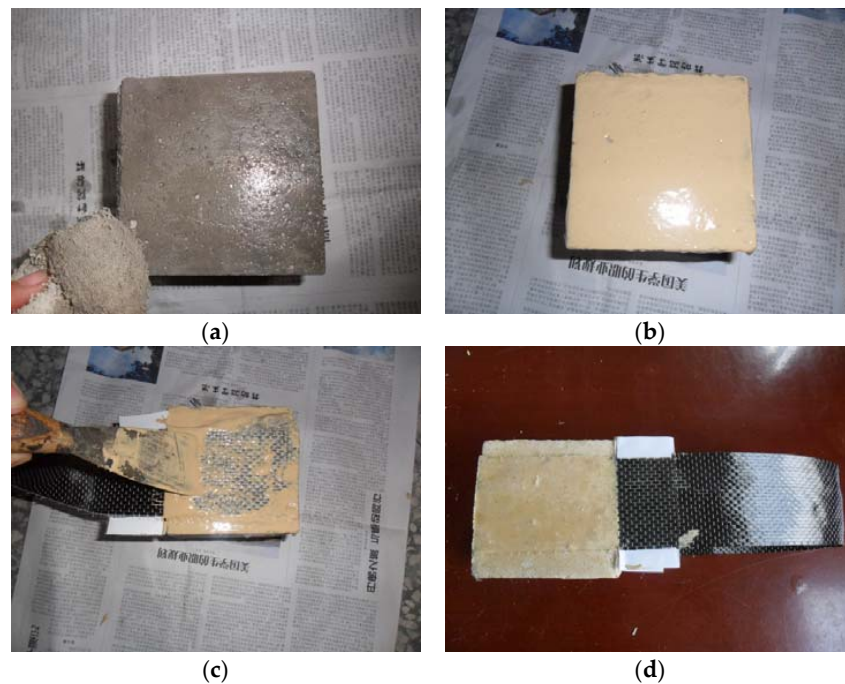


Figure 2. Process of pasting the carbon fiber sheets: (a) cleaning up the concrete surface; (b) trowelling the primer layer of magnesia-phosphate cement (MPC); (c) pasting the carbon fiber sheets; and (d) trowelling the surface layer of MPC.

The prepared specimen (Figure 3a) was kept curing for different days at room temperature and then the double-shear testing was conducted. The fixture for the double-shear test is shown in Figure 3b.

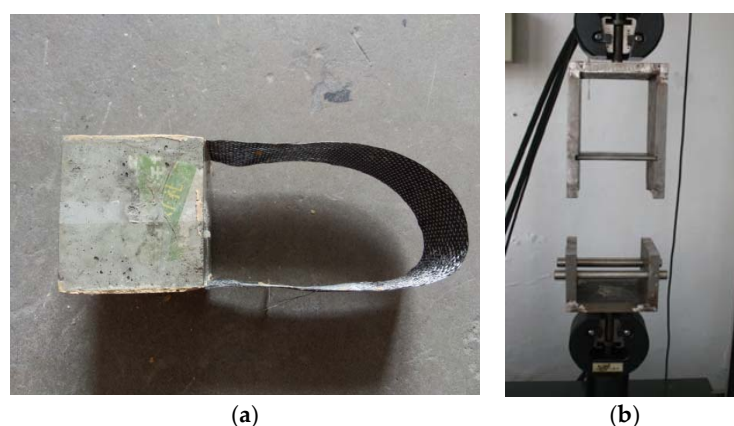


Figure 3. Specimen and fixture of the double-shear test: (a) specimen; (b) fixture.

2.2.3. Elevated Temperature Treatment

After 28 days of room temperature curing, the upper unanchored zone of carbon fiber sheets on the strengthened concrete specimens, which was not coated by MPC mixture, was wrapped with the tunnel fire coating. After the tunnel fire coating became completely dry, the specimens were firstly exposed to 105 °C in an oven for 24 h. The purpose of this treatment is to avoid explosive spalling of concrete caused by the evaporation of free water under high temperatures. Then, the dried specimens were placed in a high temperature furnace and heated up respectively to 200 °C, 300 °C, 400 °C and 500 °C for 3 h with the temperature increasing rate of 3 °C/min and then cooled to room temperature. After the high temperature treatment, the tunnel fire coating was cleaned up and then the bond strength measurement was carried out. Three specimens were tested for each mixture after every temperature exposure and the average value was evaluated as the bond strength.

2.2.4. Microscopic Characterization

Scanning Electron Microscope (SEM, FEI CO., Quanta200F, Hillsboro, TX, USA) was employed to examine the morphology and microstructure of the interfacial connection between the carbon fiber sheets and adhesives. Images were produced using the Environmental SEM in a high-vacuum mode with spot size 4.7, aperture 4 and an accelerating voltage of 20 kV. In order to observe the overall topography and microstructure of the attachment, fracture sections of all samples at room temperature were used for this analysis.

3. Results and Discussion

3.1. Failure Modes

During double-shear tests, special attention was paid to failure modes. Based on the experimental results of bond property between carbon fiber sheets and concrete surface after room temperature curing or elevated temperature exposure, several failure modes were found in this study as follows (as shown in Figure 4): (a) delamination of the carbon fiber sheets, which is the dominate failure mode under both room temperature and elevated temperature exposure lower than 200 °C in this study; (b) complete debonding failure between the adhesive and the concrete substrate, which was only occurred for several MPC specimens when the concrete has a low strength grade or the epoxy resin specimens [23]; (c) partial debonding failure between the adhesive and the concrete substrate (this failure can be attributed to minor eccentricity of loading or nonhomogeneity of adhesive matrix, or the poorer quality of concrete surface preparation) [24]; (d) failure in the adhesive layer, due to the adhesive cracking at high temperature; (e) failure at the end anchorage zones, due to minor eccentricity of loading; and (f) peeling off of the carbon fiber sheets, due to the decrease of adhesive bond strength at high temperature. The failure modes (d) and (e) have been reported in literature [25]. For the specimens failed through failure mode (a), the real bond strength cannot be obtained; therefore, the following reported bond strength for such failure specimens is lower than the real bond strength.

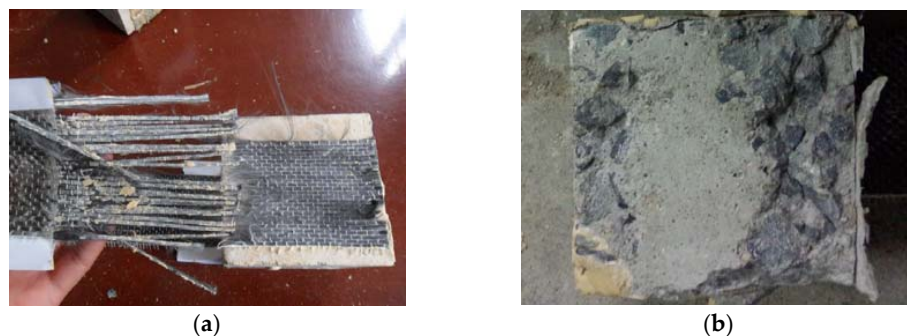


Figure 4. Cont.

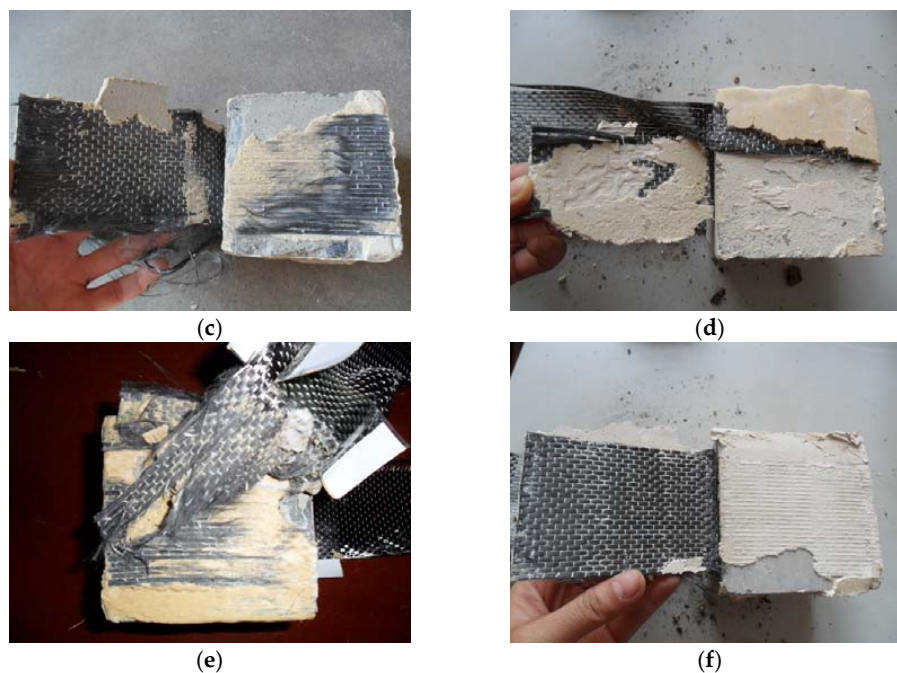


Figure 4. Typical failure modes observed from double-shear tests: (a) delamination of the carbon fiber sheets; (b) complete debonding; (c) partial debonding; (d) failure in the adhesive layer; (e) failure at the end anchorage zones; and (f) peeling off of the carbon fiber sheets.

3.2. Bond Properties at Room Temperature

For interfacial bond properties affected by types of adhesives, the used concrete specimen has a 28-day compressive strength of 30.95 MPa. The bond strength for MPC and epoxy resin specimens cured for 28 days at room temperature is shown in Figure 5. The bond strength of MPC at 28 days is much higher than that of the epoxy resin. The influence of the type of matrix has also been analysed referring to the load versus displacement, as reported in Figure 6. From Figure 6, it can be noted that the slope of MPC specimens is a little larger than that of epoxy resin specimens.

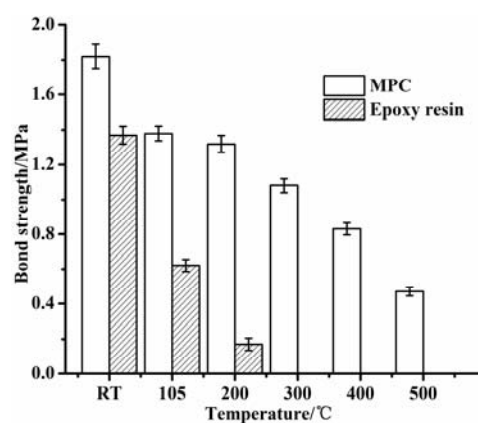


Figure 5. Bond strength of modified MPC and epoxy resin after exposure to different temperature.

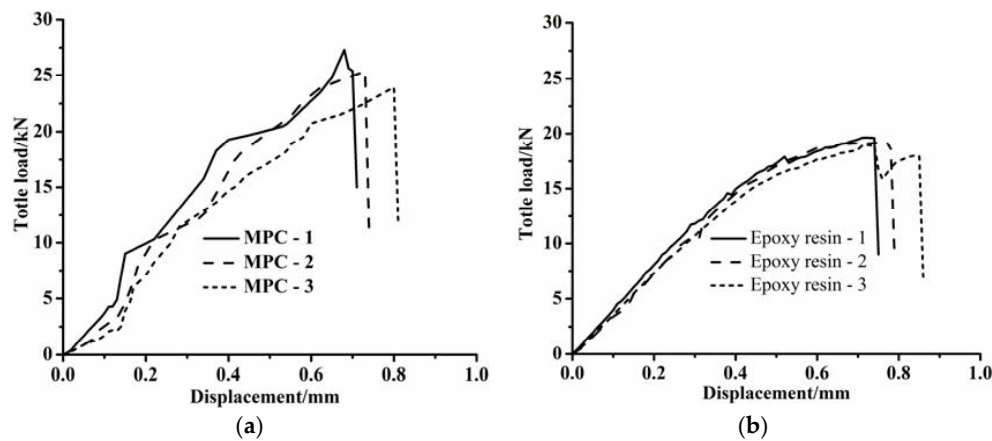


Figure 6. Load-displacement curves at room temperature: (a) MPC specimens; (b) epoxy resin specimens.

For the MPC specimens at room temperature, the failure mode is failure mode (a), perhaps due to the high viscosity of MPC mixture that failed to fully penetrate into the carbon fiber sheets. Therefore, the real bond strength of MPC should be not less than this tested value. The failure mode is failure mode (b) for the epoxy resin specimens, perhaps due to the good adhesive performance of the epoxy resin. However, the MPC specimens failed through interlaminar slip of fiber strips instead of complete debonding. MPC specimens exhibit higher bond strength than epoxy resin at ambient temperature. In addition, the bonding is very tight between the CFRP and adhesives for both the samples of the MPC and the epoxy resin cured for 28 days (see Figure 7). It may be feasible to strengthen concrete members with carbon fiber sheets bonded with the MPC mixture.

For interfacial bond properties affected by concrete strength, three different concretes were carried out with compressive strength of 30.95 MPa, 42.67 MPa and 49.89 MPa, respectively. MPC was used as the adhesive, and the curing age under room temperature was also considered. The results of bond strength are indicated in Table 3. With the increasing compressive strength of concrete substrate, the bond strength is increased slightly. This may be attributed to the fact that the higher compressive strength leads to the higher shear strength of concrete surface. The average bond strength at three days and seven days is about 82% and 93% of that at 28 days, respectively. It could be concluded that MPC has advantages of rapid hardening and high early strength.

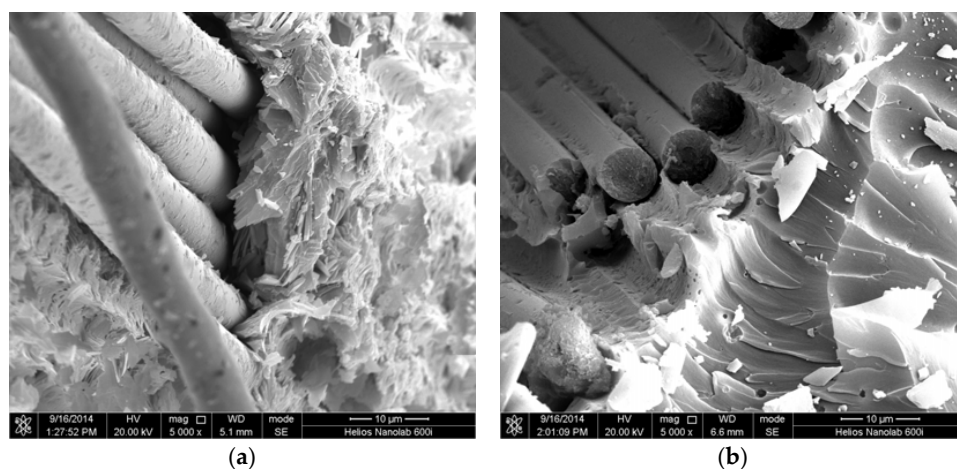


Figure 7. Interfacial connection between carbon fiber sheets and adhesives: (a) MPC; (b) epoxy resin.

Table 3. Influence of concrete strength and curing age on bond strength of magnesia-phosphate cement (MPC).

Compressive Strength of Concrete $f_{cu,m}$ (MPa)	Curing Age (Days)	Bond Strength (MPa)
30.95	3	1.50
	7	1.71
	28	1.82
42.67	3	1.61
	7	1.75
	28	1.88
49.89	3	1.68
	7	1.87
	28	2.01

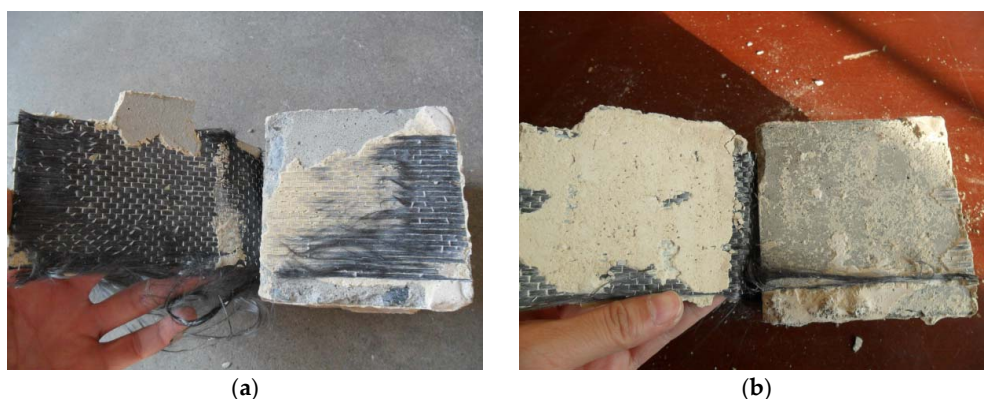
3.3. Failure Modes after Exposure to High Temperatures

3.3.1. Experimental Phenomena

The carbon fiber sheets have no obvious change when the temperature is less than 105 °C, but oxidation occurs when heating up to higher than 105 °C. The higher the temperature is, the deeper the oxidation degree is. For MPC specimens, a strong pungent smell and white smoke was emitted from the exhaust port of the furnace when the temperature was higher than 105 °C. The primary reason for this is considered to be the decomposition of hydration products of MPC. Explosive spalling of concrete specimens occurred at 500 °C for one group of MPC specimens. The epoxy resin exhibits a color of pale yellow at ambient temperature, and it gradually changes to dark yellow and ultimately black with the temperature increase. There is a burning smell when heating up to more than 105 °C, which is in accordance with the seriously degraded bond performance of epoxy resin specimens at higher temperature.

3.3.2. Failure Modes

The failure appearance of MPC and epoxy resin specimens after exposure to high temperatures is shown in Figures 8 and 9, respectively. For the MPC specimens at 105 °C, they failed through the falling-off of small lumps of concrete, perhaps due to minor eccentricities of loading or nonhomogeneity of adhesive matrices. The MPC specimens failed in the adhesive layer for all of the treatments under high temperatures ranging from 200 °C to 400 °C, perhaps due to the adhesive layer cracking. The MPC specimens failed through rupture of fiber strips, perhaps mainly due to the strength degradation of MPC or the adhesive cracking at high temperature. The complete debonding failure between the adhesive and the concrete substrate happened for the epoxy resin specimens under temperatures from room temperature to 200 °C, perhaps due to the good adhesive performance of the epoxy resin. However, peeling off of the carbon fiber sheets happened for the epoxy resin specimens at 300 °C due to the serious deterioration of epoxy resin.

**Figure 8.** Cont.

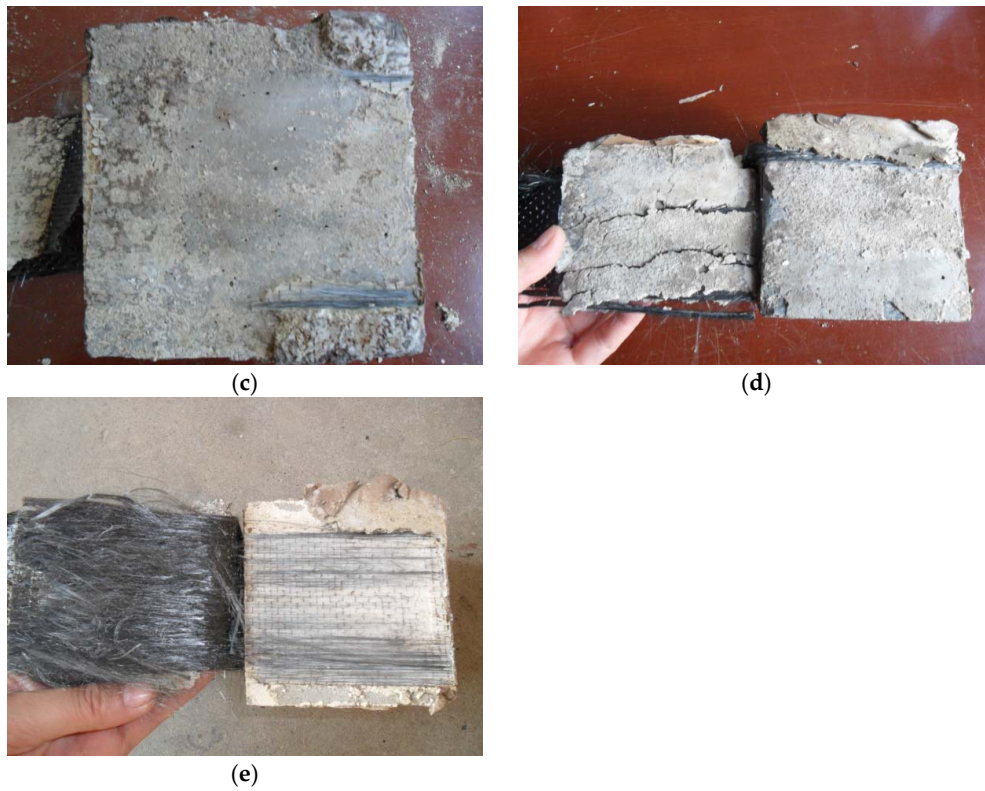


Figure 8. Failure modes of MPC specimens after exposure to high temperatures: (a) 105 °C; (b) 200 °C; (c) 300 °C; (d) 400 °C; (e) 500 °C.

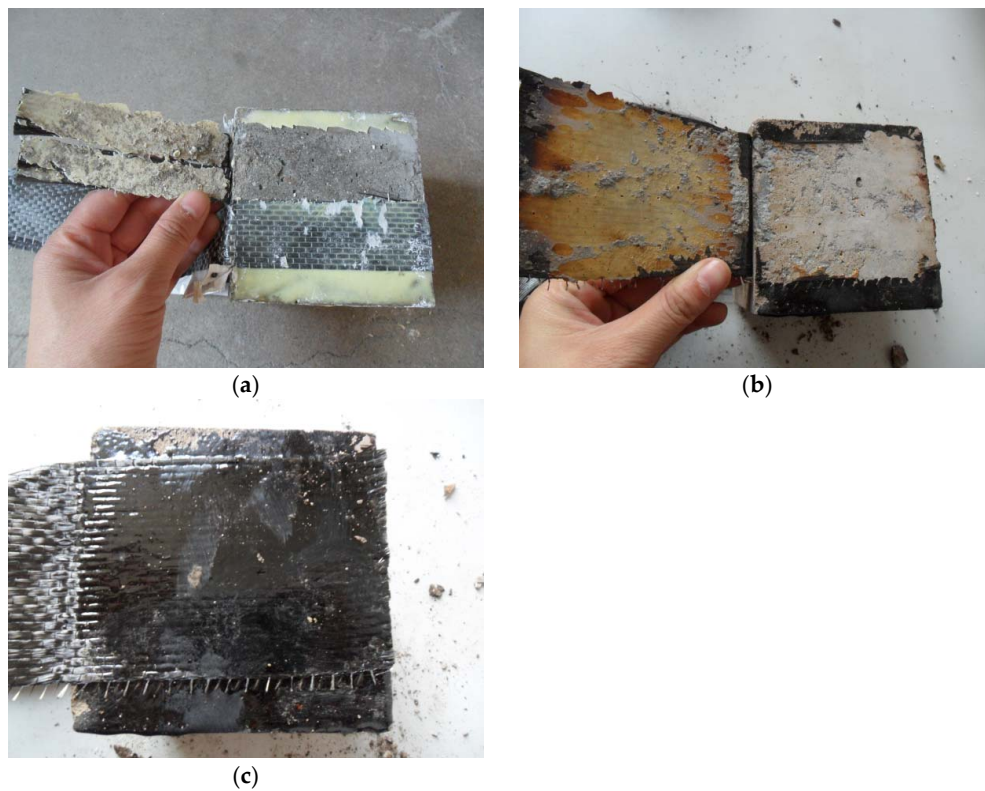


Figure 9. Failure modes of epoxy resin specimens after exposure to high temperatures: (a) 105 °C; (b) 200 °C; (c) 300 °C.

3.4. Bond Strength after Exposure to High Temperatures

3.4.1. Influence of Wollastonite Addition

Effect of wollastonite content on the bond strength of MPC exposed to different temperature is shown in Figure 10. For all the mixtures without and with the addition of wollastonite, the bond strength of MPC reduces significantly when the temperature increases to 105 °C from room temperature, but then almost remains constant from 105 °C to 200 °C. This drop in room temperature (RT)-105 °C can be mainly attributed to the loss of crystallization water of hydration products [20]. With the increasing temperature from 200 °C to 500 °C, the bond strength decreases gradually. When being compared with the room temperature samples, the exposure at a temperature of 500 °C leads to about 75% reduction in bond strength. At every temperature level, the 10% wollastonite specimen showed the highest strength. Therefore, the addition of wollastonite could help to improve the bond strength of MPC at elevated temperatures.

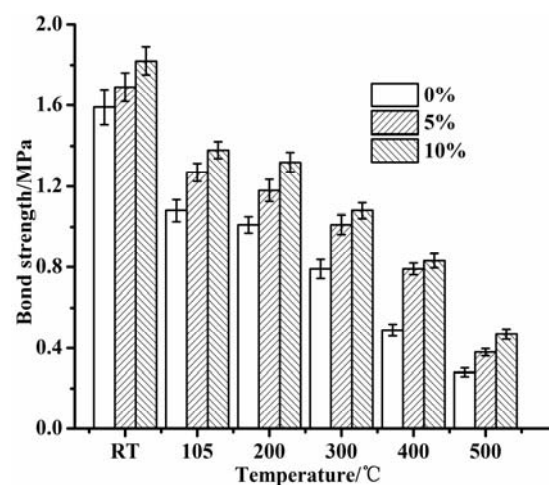


Figure 10. Effect of wollastonite content on the bond strength of MPC after exposure to different temperatures.

3.4.2. Comparison between Modified MPC and Epoxy

Double-shear tests were carried out on nine groups of specimens after exposure to temperatures from room temperature to 500 °C for evaluating the effect of temperature on bond strength of different types of adhesives (MPC and epoxy resin). The specified compressive strength of the modified MPC was 32 MPa at 28 days (the addition of 10% wollastonite). Three specimens were tested at each temperature and bond strength was evaluated as the average of bond strength obtained from these three tests. The bond strength of modified MPC and epoxy resin variation with temperature were shown in Figure 5. It can be clearly found that epoxy resin exhibits lower bond strength than MPC at ambient temperature, and retains much lower bond strength after exposures of temperatures ranging from 105 °C to 500 °C.

For the epoxy resin specimens, more than a half of bond strength was lost when the temperature increased from room temperature to 105 °C. When the temperature was increased to 200 °C, the bond strength of epoxy resin specimens decreased to 0.17 MPa, which was accompanied with serious carbonation. Full carbonation with almost 0 MPa was detected for the epoxy resin specimen after exposure to temperature of 300 °C, while only 40% reduction of bond strength was found for the MPC specimen.

The load versus displacement curves of specimens at selected temperatures are given in Figure 11. The behavior was basically linear until an abrupt load drop took place. The effect of elevated temperatures was substantial on the load-displacement curves of the epoxy resin specimens, as

shown in Figure 11c,d for 105 °C and 200 °C. The thermal exposure also affected the load-displacement curves of the MPC specimens (Figure 11a,b), while the degree of influence was relatively minor [26].

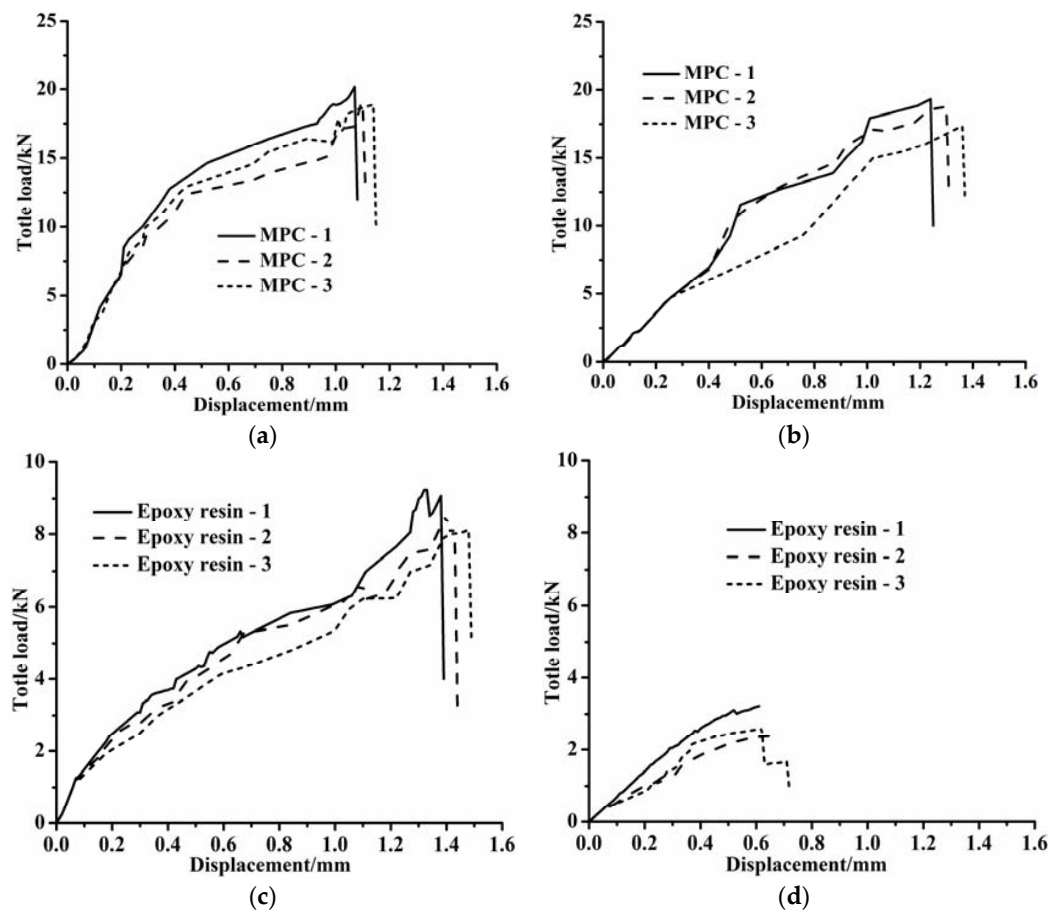


Figure 11. Load-displacement curves at high temperatures: (a) MPC at 105 °C; (b) MPC at 200 °C; (c) epoxy resin at 105 °C; (d) epoxy resin at 200 °C.

4. Conclusions

- In the double-shear test, six key failure modes occurred for concrete specimens at ambient and elevated temperatures. The delamination of carbon fiber sheet is the dominant one for the MPC specimens under temperatures below 200 °C. The MPC specimens failed in the adhesive layer at higher temperatures. The complete debonding failure between the adhesive and the concrete substrate happened for the epoxy resin specimens under temperatures lower than 200 °C. Peeling off of the carbon fiber sheet happened for the epoxy resin specimens after exposure to 300 °C.
- With the increasing compressive strength of concrete specimen, the higher bond strength between carbon fiber sheets and concrete specimen was measured. The bond strength for the MPC specimens is slightly higher than that for the epoxy resin specimen at ambient temperature, and the former reveals a much higher residual bond strength than the latter after exposure to temperatures from 105 °C to 500 °C.
- Although the MPC specimens failed through interlaminar slip of fiber strips instead of complete debonding, the improved bond strength under ambient temperature and higher temperatures revealed that the modified magnesia-phosphate cement could be a good substitute for epoxy resin in repairing and/or strengthening structural elements. Furthermore, the use of the modified MPC as the binder between the carbon fiber sheets and concrete can be less expensive and an ecologically friendly alternative.

Acknowledgments: The authors would like to acknowledge the financial support of the Program for New Century Excellent Talents in University (NCET-12-0157) (Beijing, China).

Author Contributions: Xiaojian Gao conceived and designed the experiments, and Ailian Zhang performed the experiments. Both of the authors analyzed the data and contributed to the writing of the paper.

Conflicts of Interest: The authors declare no conflict of interest.

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