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Characterizing the bond strength of geopolymers at ambient and elevated temperatures

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ABSTRACT

This paper presents characterization of bond strength of geopolymers at ambient and elevated temperatures. The bond strength of 18 different formulations of metakaolin (MK)/fly ash (FA) based geopolymers is evaluated through double shear tests in 20–300 °C temperature range. The test parameters include fly ash content, SiO_2/K_2O ratio, solid-to-liquid ratio and Si/Al ratio. In addition the effect of additives, namely short carbon fibers, basalt fibers and styrene–acrylate emulsion in MK/FA precursor, on bond strength is studied. Data from the tests show that geopolymers exhibit slightly lower bond strength than that of epoxy resin at room temperature, however geopolymers retain much higher bond strength in 100–300 °C range. Addition of small quantity of short carbon fibers in MK/FA precursor does not significantly influence bond strength of geopolymers at ambient temperature, but greatly improve bond strength retention in 100–300 °C through crack control mechanism.

1. Introduction

In the last decade, use of fiber reinforced polymers (FRP) for strengthening of concrete structures has increased significantly. The major advantages of using FRP for repair and rehabilitation of concrete structures are their high strength, low weight, corrosion resistance, and ease of application. However, the application of external bonded FRP to concrete structure is often restricted to conditions where temperatures are below the resin glass transition temperature (T_g), which is around 82 °C for commonly used organic epoxy resin [1]. This is mainly due to deterioration of thermo-mechanical properties of epoxy resin at high temperatures [2–5]. In addition, the combustible organic epoxy resin can generate flame spread and toxic smoke under fire. To overcome these concerns, substantive efforts are being made in developing fire insulating techniques for FRP strengthened structures [6,7]. Also innovative matrix/fiber systems are being developed to enhance high temperature tolerance of FRP [8]. For this purpose, recently, a new class of material, inorganic geopolymers, has been introduced as a viable alternative to organic polymers [9,10]. In comparison with organic polymers, inorganic geopolymers have advantages of resistance to high temperature, resistance to UV radiation, minimal toxic smoke under fire exposure, and ease in handling [11–13].

Research data on the adhesion at ambient temperature of fiber sheets bonded with geopolymers on concrete [8,10,11], timber [14], masonry substrate [15] has already been reported in literature. Mechanical characterization of different types of geopolymers exposed to high temperature also has been studied, but these studies mainly focused on the residual compressive and flexural strength after high temperature exposure [16–21], bending and compressive strength at high temperatures [22], and the bond properties of geopolymers with steel plate at high temperature [23,24].

Bond strength of geopolymers is highly dependent on its constituent materials, formulation and curing conditions. Although the effect of these parameters on bond strength at ambient temperature is indirectly considered in few studies [8,10,11], there is lack of data on the bond strength of geopolymers under high temperature exposure.

To develop reliable data on the effect of temperature on bond strength of geopolymers, a number of double shear tests were carried out on mortar–geopolymer–fiber sheet systems. Metakaolin (MK)/fly ash (FA) based geopolymers with different formulations are applied to bond fiber sheets and cement mortar substrate, and the effect of critical factors on bond strength of geopolymers is investigated through double shear tests at ambient and elevated

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temperatures. The studied parameters include composition and proportion of MK/FA precursor and alkaline–silicate activator, additive type and content, and curing humidity conditions.

2. Test program

The experimental program for characterizing bond strength of geopolymers consisted of a large number of double shear tests at ambient and elevated temperatures on cement mortar–geopolymer–fiber sheet systems, in which geopolymers were prepared with different formulations. Comparative tests were conducted on mortar–epoxy resin-fiber sheet systems.

2.1. Raw materials and preparation of geopolymers

The primary aluminosilicate source material used in preparing test specimens for experiments is metakaolin (MK) and fly ash (FA). Commercially produced metakaolin, supplied by Shanxi Jinkunhengye Co., Ltd., China, through calcining kaolin under 900 °C, was used in preparing test specimens. Geopolymers from pure MK is more susceptible to shrinkage [16,25–27]. To minimize such shrinkage, MK was partially substituted by replacing it with fly ash (FA) of Grade I type as per Chinese Code GB/T 1596-2005 [28] specifications. The chemical composition of MK and FA used in the mix is tabulated in Table 1. Three combinations of FA/(MK + FA) with mass ratios of 0, 0.05 and 0.1 was investigated in this study.

The alkaline–silicate activator of the desired composition were formulated by blending different proportions of commercial potassium silicate solution with 15.8 wt% K_2O, 24.2 wt% SiO_2 and 60 wt% H_2O (SiO_2/K_2O molar ratio is 2.4), and potassium hydroxide flakes with 85% purity, and tap water, to obtain desired SiO_2/K_2O molar ratios (1.0, 1.2 and 1.4) respectively. Table 2 presents mix proportions for preparing alkaline–silicate activator solution with different SiO_2/K_2O molar ratios. All activator solutions were prepared 1 day prior to use, due to high heat released by dissolving potassium hydroxide flakes. A solid-to-liquid ratio of 0.5, 0.6, 0.8 and 1.1, representing mass ratio of MK/FA precursor to alkaline activator solution, was adopted in this study.

A small amount of silica fume (superfine, 95% pure) and $Al(OH)_3$ powder (99.8% pure) was added to MK/FA mixture to obtain required molar ratio of Si/Al.

Previous studies have indicated that MK-based geopolymers are relatively brittle and prone to crack, especially under high temperature [16,27]. To improve flexibility and minimize cracking, two types of additive materials, namely short fibers and organic polymers, were added to MK/FA precursor. Short fibers are often employed as toughening agents to enhance flexural and tensile strength of geopolymer concrete [29,30], but their influence on bond strength of geopolymers is seldom studied. Two types of short fibers, including short carbon fibers (CF) and short basalt fibers (BF), were used for reinforcing geopolymers in the test program. It is reported that the incorporation of a small amount of organic polymers in geopolymers can improve compressive and flexural strength of geopolymers [20]. Thus, one type of organic polymer, styrene–acrylate emulsion (SAE), was added to geopolymers to investigate its effect on bond strength of geopolymers.

The length, diameter and density of short carbon fibers are 6 mm, 7 μm and 1.76–1.80 g/cm^3 respectively. The length, diameter and density of short basalt fibers are 6 mm, 13 μm and 2.65–3.00 g/cm^3 respectively. The solid content of styrene–acrylate emulsion is 48 ± 2 wt%, PH value is 7.5–9 and viscosity coefficient is 800–1000 mp.s. Three different mass contents of 0%, 0.5% and 2% of these additives to MK/FA precursor were considered.

Geopolymers were prepared by hand-mixing MK/FA precursor, additives (BF, CF or SAE) and alkaline silicate solution for 5 min and then mixing all the ingredients in a mixer for 10 min.

2.2. Preparation of test specimens

For undertaking double shear tests, 120 cement mortar prism blocks of size 160 × 40 × 40 mm were prepared as per Chinese Code GB/T17671-1999 [31] specifications. These blocks were cast using triplet steel moulds and cured in a tank for 28 days at 20 °C temperature and 90% humidity. The mix proportion of cement mortar is listed in Table 3. Three mortar specimens were tested to evaluate bending and compressive strength and the average bending and compressive strength of these blocks at 28 days was found to be 8.6 MPa and 49.7 MPa respectively. The remaining blocks were dried and cleaned, and prepared for double shear tests.

Two types of fiber sheets, namely carbon fiber (CF) and basalt fiber (BF) sheets were used to strengthen the above cast mortar blocks. Both types of fiber sheets were supplied by Dongguan Russia & Gold Basalt Fiber Co., Ltd. The specified tensile strength of CF and BF sheets with permeated epoxy resin were 3085 MPa and 1400 MPa respectively, and that of CF and BF sheets without permeated epoxy resin were 1972 MPa and 808 MPa respectively. Carbon and basalt fiber sheets without permeated epoxy resin were cut into strips of 390 (length) × 40 mm (wide) in advance, for use in strengthening of mortar blocks.

As depicted in Fig. 1, two areas measuring 30 × 40 mm on the upper and lower surface of a cement mortar block were cleaned and prepared as the testing zone and anchoring zone respectively. The testing zone was primed with the prepared geopolymer matrix. After the primer almost dried, one end of the fiber strip, which was preimmersed with geopolymers, was attached to the testing zone. A small roller was used to compact the fiber strip and remove any air bubbles. The anchoring zone was strengthened at the other end of the fiber strip using the same procedure as in the case of the testing zone, but the matrix was replaced by epoxy resin, which was provided by Nanjing Mankate Sci. & Tech. Co., Ltd. Subsequently, the testing zone and anchoring zone were covered with a plastic film, and the specimen was cured in a constant temperature and humidity tank for 7 days.

Curing temperature and humidity conditions directly influence final strength of geopolymer specimens [32]. Curing temperatures in the range of 50–80 °C are widely accepted values used for successful geopolymer hydration [1,10,22,23,32]. However, a full scale structure would be difficult to efficiently heat during curing process. To explore the feasibility of the use of geopolymers in in-situ strengthening applications, all specimens in this study were cured at ambient temperature. Two types of curing humidity (50% and 90%) were adopted to study its effect on bond strength.

A summary of critical factors influencing bond strength of geopolymers, considered in the test program, is tabulated in Table 4.

Table 1
Chemical compositions of metakaolin and fly ash.

Chemical	Component (%)	
	MK	FA
SiO_2	51.85	48.66
Al_2O_3	44.84	24.8
Fe_2O_3	0.98	21.1
TiO_2	0.9	–
MgO	0.48	1.1
P_2O_5	0.45	–
Na_2O	0.16	–
CaO	0.13	3.3
H_2O	0.12	–
K_2O	0.08	–
MnO	0.01	–
S_2O_3	–	1.04