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Behavior of combined fly ash/GBFS-based geopolymer concrete after exposed to elevated temperature

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Abstract. Geopolymer is an emerging cementitious material characterized by better mechanical performance and more environmentally sustainable properties than ordinary Portland cement (OPC). Previous work mainly highlighted thermal behavior of metakaolin or fly ash-based geopolymers. This paper provides experimental data of geopolymer concrete manufactured with granulated blast furnace slag (GBFS) and fly ash after high temperature exposures of 400°C, 600°C, 800°C and 1 000°C in terms of ISO834. Residual compressive strength and splitting strength, as well as the effects of standing time were examined and compared with equivalent OPC concrete findings. The results revealed that both kinds of strength improved about 14% and 9% after being exposed to 400°C, and the reduction was less than OPC concrete. Additionally, the residual compressive strength after different exposures evolved variously with prolonging of standing time. On the basis of the results, the empirical equations of residual compressive strength and splitting strength of GC are established.

1. Introduction

Geopolymer is a promising alternative to OPC, which is produced by synthesizing pozzolanic compounds or aluminosilicate minerals with high alkaline solutions [1]. These materials, such as metakaolin, fly ash, furnace slag, mine tailings etc, are mostly industrial by-products and can be easily dissolved in highly alkaline solutions at room temperature, eventually yielding a polymeric silicon-oxygen-aluminum framework [1]. Owing to the ceramic-like properties, geopolymers are believed to exhibit good fire resistance [2, 3]. Thus geopolymer concrete (GC) may acquire superior thermal behavior than OPC concrete. Geopolymers are also featured by low CO_2 emission and low energy consumption. The greenhouse gas emitted by the manufacture of geopolymers is reported to be 80-90% less than that by OPC [4]. The depletion of energy is as well estimated in the vicinity of 60% less than that consumed by OPC [5].

Fly ash is industrial waste derived from the combustion of pulverized coal in power stations. In China, 620 million tons of fly ash was produced in 2015. GBFS is also industrial by-product of iron works with an annual production of nearly 100 million tons. Both materials require no more calcination than metakaolin in the manufacturing process. GBFS and fly ash have widely been used as additives to OPC aimed at promoting the workability and mechanical performance, even providing higher early strength [6, 7]. Geopolymers based on GBFS and fly ash contain a smaller amount of calcium hydroxide than OPC pastes and are expected to demonstrate superior thermal behavior [8]. Nevertheless, most papers [9, 10] about GBFS/fly ash system focused on the ambient behavior like the influence of GBFS on the hydration and harden process of geopolymers. Limited work [8] concerning its thermal performance mostly explored the effects of the mixture proportions of geopolymer paste.

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To the author's knowledge, the analysis on GBFS/fly ash-based geopolymer concrete after elevated temperature is not found. Moreover, most existing papers paid attention to the mechanical performance of GC at elevated temperature, which does not signify behaviors in fire. Materials, obtaining thermal exposures in a fire, also need to accommodate with the certain rate at which temperature increase. Therefore, temperature curve in ISO834, most commonly adopting fire curve, was used as heating rate in this study. The main work of this investigation is to experimentally test the residual strength of GC through high temperature exposures and to propose the empirical equations derived from the results. In addition, the evolution of compressive strength with standing time is examined to analyze the effect of standing time after fire. Knowledge of this geopolymer concrete contributed to the development and applications of this auspicious material.

2. Experimental work

2.1 Materials

The GBFS was sourced from Chongqing XiangZhong Construction Material Co., Ltd. It was S95 grade with density of 2.90g/cm^3 and specific surface area of 470m^2/kg. The fly ash was F class and procured from Chongqing Hualon FA Co., Ltd.

The alkaline activator was composed of alkali silicate, hydroxide and water. The alkali silicate was sodium silicate (Na_2SiO_3) solution, which was supplied by Chongqing Jingkou chemical plant with modulus (M_s) equals to 3.1 (where M_s=SiO_2/Na_2O, SiO_2=25.72%, Na_2O=8.48%). The hydroxide solution for activator used sodium hydroxide (NaOH) flakes of 98% purity and clean tap water. M_s and water content (w/c ratio) can influence the workability of GC [2, 11, 12]. Therefore, the M_s and w/c ratio were adjusted to 1.8 and 0.44 according to trial experiments and results in [8].

The aggregates including coarse and fine aggregates were employed in saturated surface dry condition. The coarse aggregates were crushed limestone from a local quarry with size between 5-35mm and the fine aggregates were river sand with fineness modulus of 2.58.

2.2 Specimen preparation

Seventy-six samples with the size of 100×100×100mm were made in the investigation. Twenty of them were utilized to examine the compressive strength. Another 20 samples were employed to test the splitting strength. The rest 36 samples were prepared to examine compressive strength after various standing time.

Mixing procedures of GC and OPC concrete were similar, which was as following steps: (a) dry-mixing the fly ash, GBFS with fine aggregates together for 2 min, (b) adding the coarse aggregates and mixing for an additional 1min, (c) mixing the alkaline activator with the dry mixture for a final 1min, and (d) pouring the mixture into moulds in three layers of equal height with normal rodding and vibration.

Specimens were demoulded 1d after casting, watered for 28 days as moist curing, and covered with a polyethylene film to prevent moisture loss. At the end of curing, the specimens were allowed to dry 3d prior to thermal exposure.

It is critical to note that samples in this study were not subjected to high temperature curing normally reported in other similar studies, which would influence the initial period of geopolymerisation. Although fly ash-based geopolymers need thermal curing to make early strength high [2], GBFS-based geopolymers can acquire high early strength at room temperature [14]. It has also been reported that 28-day strength of geopolymers accelerating strength development at elevated temperature may deteriorate in comparison with mixtures treated at ambient or reduced temperature [13].

2.3 Elevated temperature exposure

After a 28-day curing, 68 specimens were equally divided into 4 groups and exposed to high temperatures of 400°C, 600°C, 800°C and 1 000°C respectively with an electrically-heated furnace. Other specimens were kept without disturbance in room temperature.

In order to simulate a fire condition, the furnace temperature was set to increase at the rate of ISO834 [15], and then sustain for 1h at the target temperature before it was turned off to let samples naturally cool to room temperature inside.

2.4 Test methods

The 28-day compressive strength and splitting strength were examined by a servo-hydraulic universal testing machine with a maximum capacity of 200kN. Rates of loading for the tests were 30MPa/min and 3MPa/min respectively.

3. Results and discussions

3.1 Qualitative observations

3.1.1Thermal exposure observations. Vapors escaped from the vent pipe of the furnace continuously during the heating process. Dehydration of free water and chemically bound water caused most steam, which usually sustained till 300°C and could continue when fast heating rate was utilized [16]. Water also sourced from dehydraxylation and alkali activation of un-reacted geopolymer precursors. In addition, the pore pressure of steam triggered initial minor cracks to geopolymers.

Damages of specimens by temperature were also observed as shown in Fig.1. The geopolymer concrete experienced color change, mass loss, cracks and corner breakage which became sequentially worse with the increase of temperature. The color of GC turned into red after exposed to 800°C, which was attributed to kinetic ferrihydrite-hematite phase change resulting from high iron content in fly ash [17]. The coarse aggregates after exposures of 800°C and 1 000°C changed to white and light yellow, which might be caused by decomposition of limestone aggregates after high temperature. Compared with previous work [18, 19], GC had slighter damage than OPC concrete, and no visible spalling was observed which was common in OPC concrete experiments.

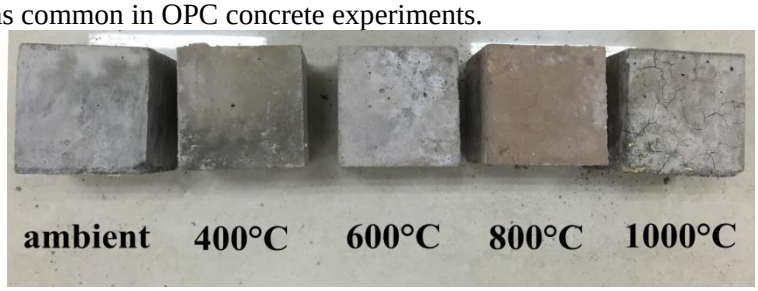


Fig 1. Surface changes of GC specimens after temperature exposure

*3.1.2Compressive failure characteristics.*As shown in Fig.2, the destruction of GC under compressive loading was opposite-cone shape with concrete breaking from the middle. The top and bottom of ambient specimen were complete for the restrictions of base plates. While more concrete peeled, the edge of the top and bottom began to crack as well with the temperature increasing.

After exposures of 800°C and 1 000°C, the coarse aggregates of crushed samples appeared without any mortar bonded, which resulted from cracks at the interfacial transition zone caused by the incompatible deformation between aggregates and geopolymer paste under heating. The failure characteristics and variation of GC resembled the results given by similar experiments of OPC concrete [18].

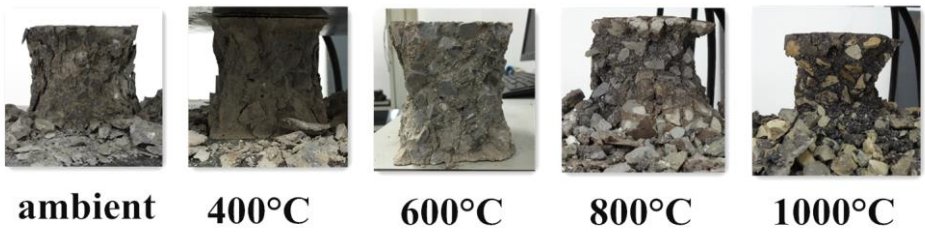


Fig 2. Failure characteristics under compressive loading

3.1.3Splitting failure characteristics. The sections of GC were presented in Fig.5. The section of the ambient sample was level and low-porous with all coarse aggregates neatly splitted and tightly bonded to mortar. Depending on fracture mechanics, cracks occurred at the weakest point and developed along the path which consumes the least energy under splitting tension. The fracture path along the interface between coarse aggregates and mortar was longer. Thus the breakage through the coarse aggregates indicated the strong bond of GC.

The sections after exposed to 400°C and 600°C were the same as the ambient specimen. However, prominent changes took place beyond 600°C. The surface layer and coarse aggregates were respectively red and grey after exposure to 800°C. While after exposed to 1 000°C, the coarse aggregates turned light yellow and the geopolymer was dark with white sand scattered inside. As the temperature rose, the sections became more porous and rough with loose texture and exposed entire stones. The geopolymer matrix would start to break down and weaken the bonding above 600°C. Moreover, incompatible deformation promoted the expansion and propagation of cracks at the interface. Consequently, the bonding between paste and aggregates was completely destroyed.

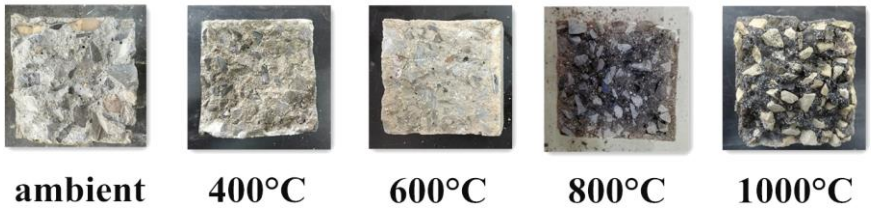


Fig 3. Failure characteristics under splitting tension

3.2 Compressive strength results

3.2.1 Residual strength. Compression behavior is one of the most principal properties of concrete, not only ranking concrete but also closely relavant to other mechanical properties. Residual strength of GC is also the major measurement to appraise the damage of structures after fire.

Ratio of ambient compressive strength of GC (f_c^T / f_c) after exposed to elevated temperature was shown in Fig.4. Value of the curve is average of experimental values from one certain group of specimens referring to Chinese standard GB50081-2002 [20]. The equivalent OPC concrete findings [21] with empirical equation and curve of the remaining compressive strength are also presented in Fig.4. Although authoritative comparison was difficult for many variations of GC and OPC concrete, the evolution of the remaining strength after high temperature was meaningfully comparable property.

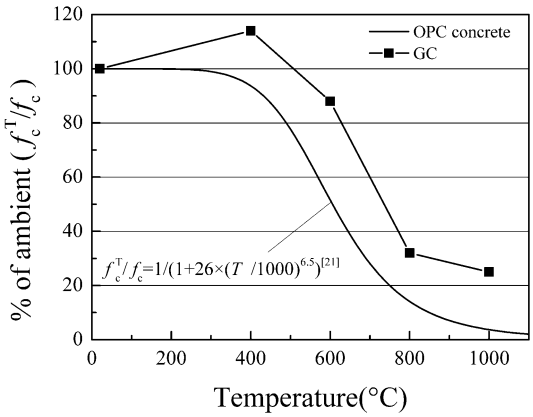


Fig 4. Residual strength of GC and OPC concrete

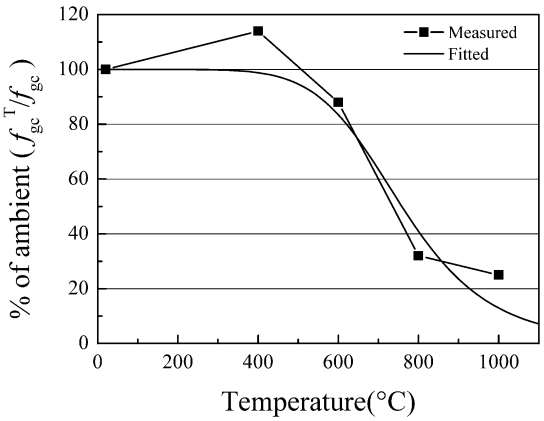


Fig 5. Fitted curve of residual compressive strength

Results in Fig.4 implied that the remaining compressive strength obviously improved about 14% after exposed to 400°C. The geopolymerisation of geopolymer precursors catalyzed in heating contributed to the strength behavior when free water was not liberated. The incompletion of initial alkali activation led to a large amount of remnant particles as well owing to ambient curing. The strength gain behavior of geopolymer and its composite after temperature exposure was common and also reported in other literatures [2, 22-24]. Additionally, the minor cracks caused by vapor pressure and thermal incompatibility did not evidently cripple the strength of GC. The geopolymerisation process predominated over the initial damage, which drew up a net result of strength gain in GC. However, this strengthening effect was complicated which might lie in mixture composition and quality of raw materials.

The residual strength reduced to 88% of the ambient after exposure to 600°C. The geopolymerisation ceased due to the loss of moisture while the densification of geopolymers and the sintering of un-reacted material occurred and might last under 900°C [16]. Densification could fill the voids and cracks, leading to greater strength. Sintering provided increased strength for strengthening the bonding between particles. Meanwhile, the geopolymer matrix was still completed [21, 25]. Therefore, the main cause of the reduction was likely to be the thermal incompatibility between paste and aggregates [26] which usually comprised between 65% and 80%. Previous studies [16, 26, 27] revealed that while coarse aggregates continuously expanded at elevated temperature, geopolymers underwent substantial shrinkage. Besides, the dehydration and dehydroxylation might be partly responsible for the reduction of strength [24].

After exposure to 800°C, compressive strength declined sharply to 32% of ambient. The geopolymer paste might decompose to crystallize to various zeolitic phases and alkali feldspars and pronouncedly crippled the strength of GC [16]. Additionally, the incompatible deformation was increasingly enhanced and caused linked cracks at the interface of GC.

GC specimens were exposed to 1 000°C and collapsed with 25% strength of ambient. The strength of paste decreased continuously. Moreover, the incompatible deformation could not be sustained any longer. The disintegration of GC commenced as a result of the initiation and propagation of cracks.

The evolution of residual properties of GC was found superior to that of OPC concrete, which lost more strength at the same temperature. Comparatively, GC was characterized by the thermal strengthen gain behavior and less reduction of strength. Furthermore, the descending branches of the two curves were comparable. Accordingly, the equation of OPC concrete was utilized to fit the reducing residual strength evolution of GC through non-linear square method with MATLAB. The empirical equation was shown in Eq. (1).

$$\frac{f_{gc}^{T}}{f_{gc}}=\frac{1}{1+6.7\left(\frac{T}{1000}\right)^{6.9}} \quad (1)$$

Where f_{gc}^{T} is the residual compressive strength of GC after temperature exposure, f_{gc} is the ambient compressive strength of GC. Fig.5 described that the fitted equation neglected the strengthening effect and conformed with the measured values, which could be applied to practical engineering.

3.2.2Effect of standing time. It was announced in [28, 31] that compressive strength of OPC concrete after fire would continuously decrease standing in air. Correspondingly, it was essential to investigate the GC's behavior under different standing time, which was important for transferring material and reinforcing structures after fire.

With the standing time of 1d, 3d, 7d and 28d, the residual compressive strength of GC was recorded in Fig.6. It revealed that the standing time had diverse influences on GC after various temperature exposures, which were associated with the chemical and physical changes in the course of heating process and standing period.

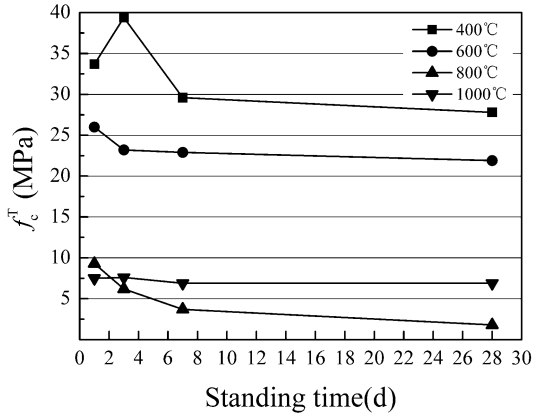


Fig.6 Residual compressive strength of GC with standing time

As analyzed above, the pore pressure, chemical changes and thermal incompatibility were grounds for the failure of GC under elevated temperature. In accordance with current studies [30, 31], there would be three changes during the cooling and standing period: (a) Cracks occurred at the interface at the early stage of cooling process resulting from the non-uniform thermal field, but cracks might recover after cooling to room temperature. (b) Moisture in air contacted remnant geopolymer precursors through surface cracks and might activate geopolymerisation reaction. (c) Burned-out coarse aggregates would absorb water and expand, which caused interior tension stress inside GC. These three changes occurred to various extents in all specimens.

Residual strength of GC after exposed to 400°C came to peak at 3d, decreased significantly at 7d and then reduced slightly at 28d. The continuation of geopolymerisation contributed to the strength gain behavior at 3d, while other damages dominated with the prolonging of standing time. Specimens exposed to 600°C and 800°C had similar monotonous decrease of residual strength. The sintering of raw materials restricted the further hydration which might lead to strength gain. The residual strength of specimen after exposure to 1 000°C seldom changed with standing time. The incompatible deformation developed fully and coarse aggregates deteriorated, where the expansion of aggregate or strengthening behavior was almost not evident.

Above all, the general trend of residual strength flattened beyond 7d with a total trend of decrease. Accordingly, the structures made with GC should be repaired at once after fire. Further investigation should be carried out for the influence of the combined multiple factors.

3.3 Splitting strength results

The tensile strength of concrete is normally low, which is not directly utilized except for some special elements and pre-stressed concrete. However, it can not be neglected owing to the close relativity to crack resistance, shear capacity and torsion bearing capacity of concrete elements. The splitting strength of GC in this study was used to demonstrate the tensile strength.

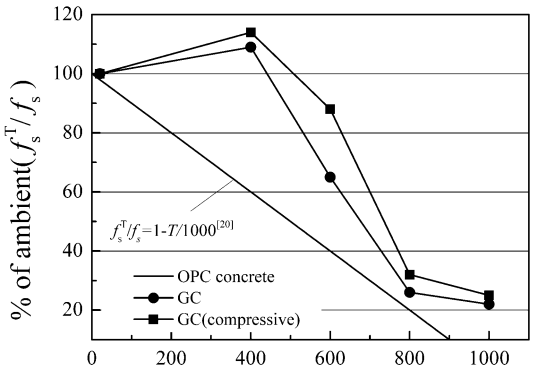


Fig 7. Residual properties of GC

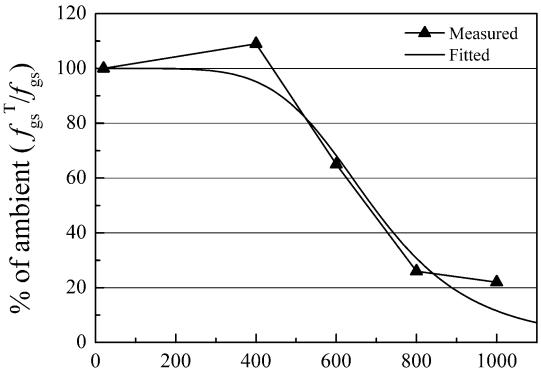


Fig 8. Fitted curve for residual splitting strength of GC

Ratio of ambient splitting strength (f_s^T / f_s) and the results of compressive strength were illustrated in Fig.7. Consistent with the results in section 3.2.1, the splitting strength also increased by 9% after exposure of 400°C. The curves elucidated that the splitting and compressive strength more or less adopted a similar trend, while the splitting strength revealed a higher rate of decline. The decline of residual splitting strength of OPC concrete was approximately linear [21], which could be summarized as the segment line and the equation in Fig.7. Utilizing the fitting method of Eq. (1), the empirical equation of the variation trend of GC's residual splitting strength was presented as Eq. (2).

$$\frac{f_{gs}^T}{f_{gs}} = \frac{1}{1+7.7\left(\frac{T}{1000}\right)^{5.5}} \tag{2}$$

Where f_{gs}^T is the residual splitting strength of GC after thermal exposure, f_{gs} is the ambient splitting strength of GC. As shown in Fig.8, disregarding enhancement by the reason of safety, the fitted curve accords with the splitting measurements and can be applied to practical engineering.

4. Conclusions

The present study adds new data to the fire behavior of geopolymer concrete synthesized with GBFS after exposures to 400°C, 600°C, 800°C and 1 000°C respectively at rate of ISO834 fire curve. The research objects are remaining compressive strength, splitting strength of GC and the effects of standing time on compressive strength. The experimental results led to the following conclusions.

(1) Geopolymer concrete had obvious surface changes after exposed to elevated temperature, which was superior to OPC concrete and could be used to appraise the thermal damage of structures.

(2) There were strength gain behaviors of residual compressive and splitting strength, which could increase by about 14% and 9% respectively. The compressive and splitting strength had a similar trend of reduction. Both trends were also corresponding to the compressive strength evolution of OPC concrete, but definitely differed from the rule of splitting strength. The empirical equations were presented which accorded with the measure values and can be put into applications.

(3)Under the coupling effects of high temperature exposures and standing time, the residual compressive strength of GC changed variously, which was mainly due to the chemical and physical

changes in the course of heating process and standing time. Nevertheless, the residual strength of all specimens became steady 7 days later.

In addition, Evolution of microscopic characteristics including microstructure change and phase change of fly ash/GBFS-based geopolymers exposed to elevated temperature should be investigated further.

Acknowledgement
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