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# Fiber Reinforced Geopolymers for Fire Resistance Applications

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## Abstract

This paper presents the development of fiber reinforced metakaolin-fly ash based geopolymers for fire resistance application. A series of experiments are carried out to develop optimum mix proportions of fiber reinforced metakaolin-fly ash based geopolymers with superior fire resistance properties. Bending and compression tests were conducted at ambient temperature and after exposure to elevated temperatures on geopolymers with different proportion of carbon fiber and fly ash substitution for metakaolin, and the effect of carbon fiber and fly ash content were quantified. Data from the tests showed that the addition of chopped carbon fibers in geopolymers provides effective crack control and enhances bending strength under high temperature. Further partial substitution of fly ash for metakaolin reduces the water demand in the reaction process on geopolymer preparation and microstructure damage under high temperatures due to evaporation of water present in geopolymers, thus enhance the mechanical properties of geopolymers after exposure to elevated temperatures. Based on these experiments, a geopolymer made with 50% metakaolin and 50% fly ash and reinforced by 2% chopped carbon fibers can be an effective alternative material for structures in fire resistance applications.

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*Keywords:* metakaolin, fly ash, carbon fiber, geopolymer, high temperature

## 1. Introduction

Portland cement is the most widely used construction materials for decades due to its excellent thermal performance, mechanical properties and durability. However, there is a growing debate in environmental circle on the tremendous energy consumption and emission of carbon dioxide during its production. With the mounting pressure to reduce energy consumption and pollution in all industrial processes in global manufacturing, the pursuit of alternative cementitious material with less energy consumption and pollution has become an important objective of many recent research studies.

Geopolymer is a new environment friendly inorganic binder. It has attracted spotlight over the past decade with its comparable performance with Portland cement and is regarded as a most promising alternative to Portland cement [1]. Geopolymers can be produced by alkaline solution activating aluminosilicate source material, such as metakaolin (MK) [2], fly ash (FA) [3] and slag [4]. Among these materials, metakaolin is commonly used due to less impurities and good mechanical properties exhibited on MK-based geopolymers. However, it is reported that MK-based geopolymers exhibit brittle response similar to ceramics, and are susceptible to thermal cracking and rapid strength degradation at high temperature [5-8]. This is due to the high water demand during the reaction process of MK, which affects the stability and strength development of geopolymers under high temperatures [5]. To deal with these concerns, several studies explored the addition of chopped fibers [9], organic resin [10], granulated slag [5], fly ash [11] and other materials as reinforcement

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agent in geopolymers. However, these studies mainly focused on characterizing mechanical properties of reinforced geopolymers at ambient temperature. There is a lack of data on the mechanical properties, especially bending strength, of reinforced geopolymers after exposure to high temperatures. Such data is essential for evaluating fire performance of structural member, which is a basis design requirement in building.

To develop property data on fiber reinforced MK-based geopolymers, a series of experiments were carried out at ambient and elevated temperatures. Chopped carbon fibers are selected as reinforcement agent and added into metakaolin precursor to provide crack control and enhance bending strength; also metakaolin is partially replaced by fly ash to reduce the water demand during reaction process and improve high temperature performance of geopolymers. The bending and compressive strength of carbon fiber reinforced geopolymers made from metakaolin and fly ash were tested at ambient temperature and after exposure to elevated temperatures, and the effect of carbon fiber and fly ash content on bending and compressive strength of geopolymers are evaluated.

## 2. Test Program

The experimental program is composed of a large number of bending and compression tests on geopolymers with different carbon fiber content and substitution of fly ash for metakaolin at ambient temperature and after exposure to elevated temperatures.

### 2.1. Raw materials

The primary aluminosilicate source material used in preparing specimens for property tests is metakaolin (MK) and fly ash (FA) mixture. Commercially produced metakaolin with an average particle size of 0.017 mm, was supplied by Shanxi Jinkunhengye Ltd., China, through calcining kaolin under 900°C. Low calcium fly ash, with an average particle size of 0.032 mm, was supplied by Guangzhou Huangpu Power Plant. Potassium silicate solution with SiO<sub>2</sub>/K<sub>2</sub>O molar ratios of 1.0 was used as alkaline-silicate activator. Chopped carbon fibers (CF) were added to MK-FA blend precursor as reinforcement agent. The length, diameter and density of chopped carbon fibers are 6 mm, 7 μm and 1.76-1.80 g/cm<sup>3</sup> respectively.

### 2.2. Preparation of specimens

Geopolymer specimens with different content of chopped CF and FA were prepared for undertaking bending and compression tests. Five combinations of FA/(MK+FA) with mass ratios of 0, 20%, 50%, 75% and 100% was investigated in this study. And four different mass contents of 0, 0.5%, 1% and 2% of chopped CF to MK-FA precursor were considered.

The geopolymers with lower FA and high MK content has higher liquid demand to achieve similar workability than those with higher FA and lower MK content, due to fly ash having spherical shape particles and larger particle size as compared to MK which has plate-like structure and finer particle size [8]. In this study, a solid-to-liquid ratio of 0.8 and 1.1, representing mass ratio of MK-FA blend precursor to alkaline activator solution, was adopted for geopolymers with lower FA content (0% and 20%) and higher FA content (50%, 75% and 100%) respectively.

The geopolymers were prepared by hand-mixing MK-FA precursor and chopped fibers for 2 minutes, then adding alkaline silicate solution and mixing all ingredients in a batch mixer for 12 minutes. Subsequently, geopolymers were cast into a triplet steel mould with each specimen size of 160×40×40 mm and then vibrated on a shake table to remove any air bubbles. After covering with plastic films, specimens were cured for 7 days in a tank at a constant 22°C temperature and 95% humidity, and then taken out to dry naturally in a room for 1 day before testing.

A total of 75 specimens grouped into 8 sets, with different combinations of FA and CF content, were prepared for bending and compression tests. Table 1 lists the detailed mix proportions and test temperatures of these specimens. The designation GP is used for denoting geopolymer, and the numbers before and after “hyphen” symbol represent the content of FA and CF respectively. Three specimens are tested for each group at each temperature.

Table 1. Mix proportions of geopolymers and target temperatures for mechanical property tests

Group No.	FA/(FA+MK)	CF/(FA+MK)	solid-to-liquid ratio	Temperature (°C)
GP00-0	0	0	0.8	25, 500
GP00-0.5	0	0.5%	0.8	25, 100, 200, 300, 400, 500
GP00-1	0	1%	0.8	25, 500
GP00-2	0	2%	0.8	25, 100, 200, 300, 500, 600, 700

GP20-2	20%	2%	0.8	25, 500
GP50-2	50%	2%	1.1	25, 500
GP75-2	75%	2%	1.1	25, 500
GP100-2	100%	2%	1.1	25, 500

### 2.3. Test apparatus and procedure

The bending tests at ambient temperature were conducted as per Chinese Code GB/T17671-1999 specifications [12], using an universal material testing machine RGL-20A. The central distance between two supporting ends of a specimen was adjusted to 100 mm. The load was applied at the mid-span of the specimen, and increased at a rate of 50 N/s, until the specimen split into two parts. Then a compression test was carried out through CMT51005 universal material testing machine on each of the two broken parts separately. The compression area was 40×40 mm, and the loading velocity was kept at 2500 N/s.

For undertaking bending and compression tests on temperature exposed specimens, the specimens were first heated to target temperature in an electrical furnace, at a heating rate of 5°C per minute. Once the predetermined target temperature was attained, specimens were kept at the peak temperature for 60 minutes to attain thermal stability. Then the heating in furnace was turned off and the specimens were allowed to cool naturally. After cooling down to ambient temperature, the specimens were taken out of the furnace and then bending and compression tests were conducted.

## 3. Results and Discussion

Data generated from bending and compression tests of geopolymers with different CF and FA content are used to quantify the effect of CF and FA on bending and compressive strength of MK-FA based geopolymers.

### 3.1. Effect of carbon fiber content

Fig. 1 presents the pictures of MK-based geopolymer specimens (GP00-0 and GP00-2) exposed to 500°C. It can be seen that MK-based geopolymers without carbon fibers (GP00-0) develop serious cracks, and is highly prone to rupture even under smaller loads. However, no noticeable cracks develop in the specimens with 2% carbon fibers (GP00-2). This clearly infers that chopped carbon fibers can provide effective crack control mechanism in geopolymers under high temperature.

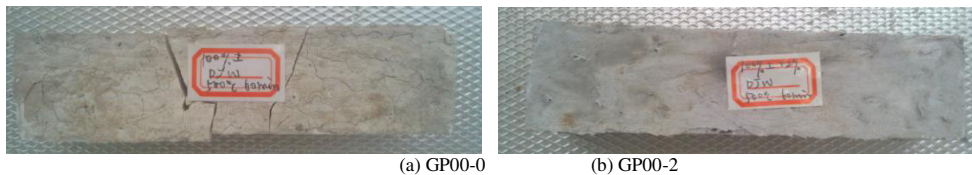


Fig. 1. Cracking in geopolymer specimens with (2%) and without CF after exposure to 500°C

The bending and compressive strength of MK-based geopolymers with different CF content (Group GP00-0 to GP00-2.0) at ambient temperature and after exposure to 500°C is plotted in Fig. 2. It can be seen in Fig. 2 that bending strength of geopolymers at ambient temperature gets significantly enhanced with an increase in CF content, but the compressive strength at ambient temperature is not affected to a great extent. After exposure to 500°C, bending and compressive strength decreases greatly for all specimens, though the specimens with higher CF content exhibit relative higher strength retention.

To investigate the variation of strength of geopolymers with temperature, bending and compression tests were supplemented on GP00-0.5 and GP00-2.0 specimens after exposure to various temperatures and results are plotted as a function of temperature in Fig. 3. It can be clearly seen in Fig. 3 (a) that the bending strength of GP00-0.5 and GP00-2.0 increases slightly at 100°C, and then slightly decrease at 200°C, thus the bending strength of specimens after exposure to 200°C is close to that at ambient temperature. However, rapid deterioration in bending strength occurs at 300°C and 500°C and beyond 500°C there is very little strength left. Comparing bending strength of GP00-0.5 and GP00-2.0 at different temperatures, it can be seen that geopolymers with higher CF content exhibit higher bending strength throughout the temperature range, but the strength difference between the two groups of specimens decreases with temperature, and approaches zero at 500°C. Accordingly, it is inferred that the reinforcing effect of CF on bending strength of geopolymers diminishes with increase in temperatures. It is reported that the tensile strength of carbon fibers decreases with increase in

temperature, and only retains 25% of ambient temperature strength after exposure to 500 °C [13] . Thus, the addition of CF almost has no influence on bending strength of geopolymers after exposure to 500 °C.

The variation of compressive strength of GP00-0.5 and GP00-2.0 with temperature is similar, as shown in Fig.3 (b), which indicates that the addition of carbon fiber has no noticeable effect on compressive strength both at ambient temperature and after exposure to elevated temperatures.

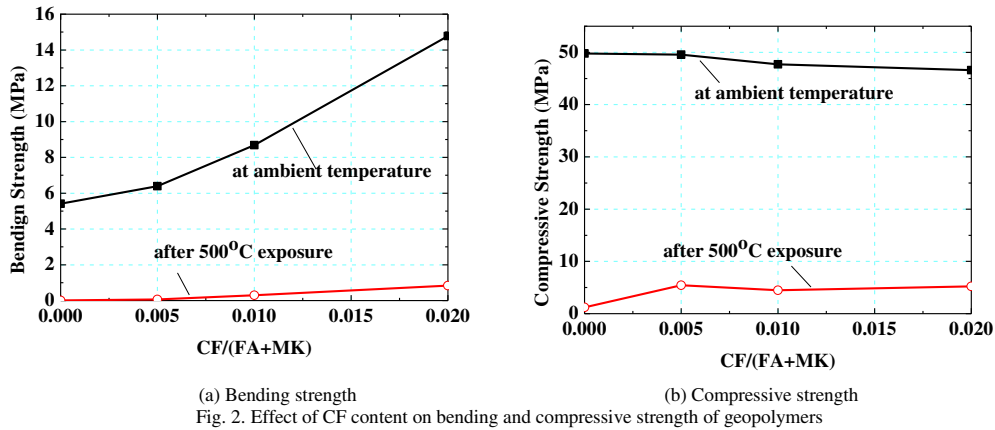


Fig. 2. Effect of CF content on bending and compressive strength of geopolymers

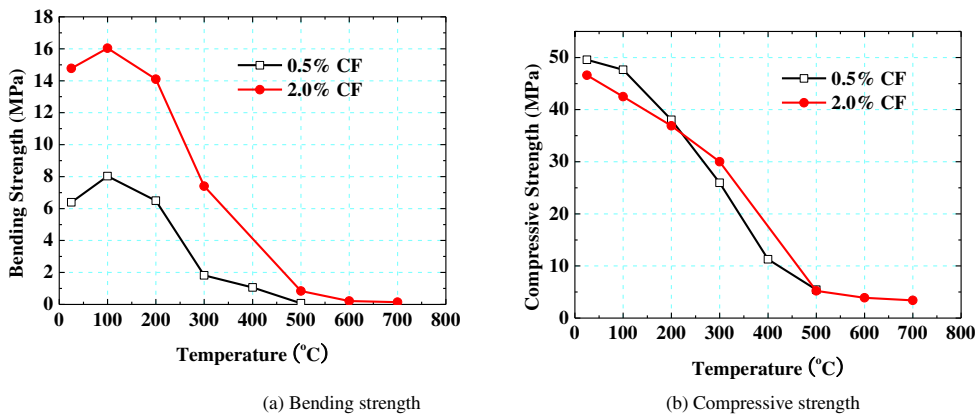


Fig. 3. Effect of CF content on bending and compressive strength of geopolymers

### 3.2. Effect of fly ash content

The test results of geopolymer with different FA content, but a constant CF content of 2% are presented in Fig. 4. From the curves at ambient temperature in Fig. 4, it can be seen that MK-based geopolymers (0% FA) possess higher bending strength at ambient temperature, but lower compressive strength as compared with MK-FA based geopolymers (20%, 50% and 75% FA). High bending and compressive strength are both achieved in MK-FA based geopolymers (20%, 50% and 75% FA), while FA-based geopolymers (100% FA) exhibit the lowest bending and compressive strength at ambient temperature. These results on compressive strength of geopolymers are in line with that reported by Rajamma et al. [14] and Fernández-Jiménez et al [11]. It was inferred in literature [14] that fly ash particles in 100% FA system is poorly dissolved during reaction process achieved by alkaline activation, and this leads to high porosity, which in turn results in low strength of FA-based geopolymers. However, in the case of MK-FA based geopolymers, the microstructure appears denser, which enhances the mechanical strength of the mixtures [14].

Comparing the strength of geopolymers at ambient temperature and after exposure to 500°C in Fig. 4, it can be seen that most of bending and compressive strength is lost after exposure to 500°C in specimens with lower FA content (0% and 20% FA), while specimens with higher FA content (50%, 75% and 100% FA) retain higher strength. The highest retention in bending and compressive strength after exposure to 500°C was achieved in specimens with 50% FA.

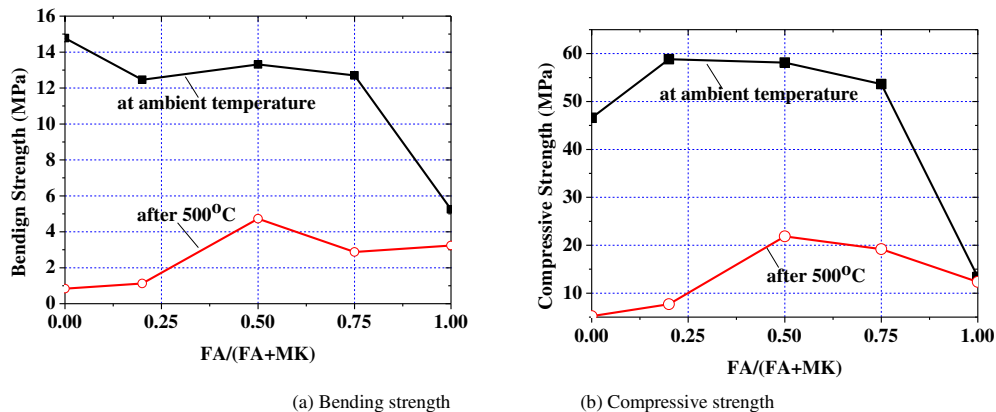


Fig. 4. Effect of FA content on bending and compressive strength of geopolymer

It is reported in literature [11, 15] that the degree of reaction achieved by alkaline activation in MK geopolymer is greater than that in MK-FA blend systems. And metakaolin reacts almost in its entirety, whereas in the fly ash, only the glassy phase reacts and may not do so fully. These un-reacted fly ash particles may occur sintering reactions at high temperature, which partly attributed to the high strength retention in geopolymers with high content of FA [8].

Another explanation on the variation of mechanical properties of geopolymers with FA content is the different mass loss due to evaporation of water present in geopolymers. As temperature increases, dehydration reaction occurs in geopolymers, and moisture within geopolymers mitigates toward the surface of the specimen and escapes, which leads to the internal damage of microstructure and consequently the strength degradation of geopolymers [8]. Since the solid-to-liquid ratio of geopolymers with lower FA content (0% and 20%) is 0.8, while that of geopolymers with higher FA content (50%, 75% and 100%) is 1.1, there was less moisture in geopolymers with higher fly ash content, thus lower mass loss experienced due to evaporation of water, which in turn leads to higher retention in strength of geopolymers with higher FA content after exposure to elevated temperatures.

As depicted in Fig. 4, geopolymers made with 50% metakaolin and 50% fly ash and reinforced by 2% carbon fibers exhibit favorable bending and compressive strength both at ambient temperature and after high temperature exposure, thus it is suitable for structural members required for good fire performance.

#### 4. Conclusions

A large number of bending and compression tests were conducted on geopolymers with different formulations at ambient temperature and after exposure to high temperature in this study. Based on the test results, the following conclusions can be drawn:

- The addition of chopped carbon fibers in geopolymers provides effective crack control mechanism under high temperature exposure and this in turn enhances bending strength in 20-500°C temperature range.
- The addition of chopped carbon fibers in geopolymers does not significantly influence compressive strength of geopolymers in 20-700°C temperature range.
- Fly ash-based geopolymers, cured at ambient temperature, provide lower strength at ambient temperature compared to metakaolin-based geopolymers. However, geopolymers with higher fly ash and lower metakaolin content exhibit greater strength retention rate after exposure to high temperature, due to lower mass loss and sintering reactions of un-reacted fly ash at high temperatures.
- Geopolymer consisting of 50% fly ash and 50% metakaolin and 2% carbon fibers provides optimum bending and compressive strength properties at ambient temperature and after exposure to high temperature, and it is suited for fire resistance applications.

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