PROPERTIES OF FLY ASH BASED GEOPOLYMER CONCRETE MADE USING SPECIAL FLY ASH AGGREGATES

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ABSTRACT

The superior performance of Geopolymer paste mortar, and Concrete (GPC) when exposed to high temperatures has been well documented. However, limitations exist due to the use of similar aggregates, coarse and fine, in both Ordinary Portland Cement Concrete (OPC) and GPC. In this research GPC is produced using lightweight aggregates made entirely from Fly Ash (named FLASHAG) as replacement for ordinarily used aggregates. The current research looks into the ambient and residual properties of GPC made using these special aggregates to high temperatures of up to 1000°C. Mechanical properties including compressive strength, splitting strength, and modulus of elasticity of such concretes are reported. Comparisons with GPC produced using ordinary aggregates are made and are also compared with results reported for OPC concretes. Although at ambient temperatures all three concretes have similar properties, the performance of GPC made from FLASHAG is superior at elevated temperatures to both OPC and GPC made from ordinary aggregates.

Keywords: Geopolymer Concrete, Stress-Strain Curves, Modulus of Elasticity, Lightweight GPC

INTRODUCTION

Alkali liquids (usually a soluble metal hydro-oxide and/or alkali silicate) can be used to react with silica (SiO_2) and alumina (Al_2O_3) rich natural materials, like metakaolin or with industrial by-products, like Fly Ash (FA), Silica Fume (SF), Rice Husk Ash (RHA) or Slag to produce binders [1-3]. Such binders mixed with typical coarse and fine aggregates can form concrete, usually known as alkali activated concrete or

Geopolymer Concrete, with mechanical and thermal properties comparable or even superior to ordinary concrete. The term “Geopolymers” describes the product of the chemical reaction between the aluminosilicates and the alkaline liquid. This term was coined to represent polymerization resulting from a source material of geological origin. Although other researchers prefer using the term alkali activated FA (AAFA) when FA is used as a source of aluminosilicates, alkali activated Slag when Slag is the source of aluminosilicates, and so on [4]. Although the binders used in this research are similar to those used in previous works, this research investigates using aggregates different to the traditional natural aggregates. The aggregates used here are lightweight aggregates manufactured entirely from fly ash. Their advantages on the performance of OPC concrete have been previously reported [5]. These aggregates (given the name FLASHAG), being entirely made from fly ash have been expected to perform in synergy with the geopolymer binder. This development has been expected to enhance the performance of concrete as well as benefit the environment by pushing the utilization of fly ash to further limits.

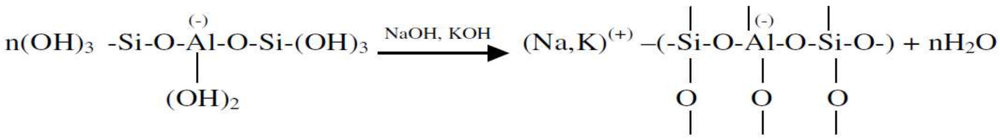
NATURE AND CHARACTERISTICS OF GEOPOLYMER CONCRETE

Though details are still debated, many researchers agree that the basic reaction mechanism is in three stages namely: dissolution of Si and Al from the source material, hydrolysis or gelation, and condensation forming a 3D network of silico-aluminates also termed as the ‘geopolymer backbone’ [1, 2, 4, 6-9]. Davidovits describes geopolymerization as an exothermic reaction, and has schematised it using equation given below [2]. GPC does not require any hydraulic binder. The role of FA in GPC is entirely different from that it plays when used as a cement replacement material in OPC concrete to enhance certain properties such as workability or to reduce the heat of hydration. In such cases FA has no pronounced effect on the strength of concrete [10] especially early strength. However, in GPC Fly Ash is the sole source of aluminosilicates for reaction with the alkaline solution to form the binder, and is thus a critical factor in strength development. The following two-stage chemical equation represents the development of the geopolymer as suggested by Davidovits [2] reported

Si-Al source + Silicates + Water + Alkaline Liquid → Geopolymer Precursor

$$(Si_2O_5,\ Al_2O_2)n + nSiO_2 + nH_2O \xrightarrow{NaOH,\ KOH} n(OH)_3\ \text{-Si-O-}\overset{(-)}{\underset{\underset{(OH)_2}{|}}{Al}}\text{-O-Si-}(OH)_3$$

Geopolymer Precursor + Alkaline Ions → Geopolymer Backbone



(1.1)

OPC AND GPC CONCRETE BEHAVIOUR AT HIGH TEMPERATURES

Owing to its very different chemistry as compared to OPC, GPC behaves very differently when exposed to high temperatures. When concrete is taken to high temperatures, the change in the concrete properties are highly irreversible [11]. It is widely accepted that OPC loses over 80% of strength when the temperature exceeds 800^{o}C. This is due to inherent physical stresses caused by the introduction of heat energy and the chemical breakdown of the hydrated phases in the cement paste [12]. There have been a number of general findings published in the literature that cover the effects of high temperature on OPC concrete, though the extent may depend on several factors like water-cement ratio, curing condition, aggregate type and size, type of admixture, and so on. These are summarised as:

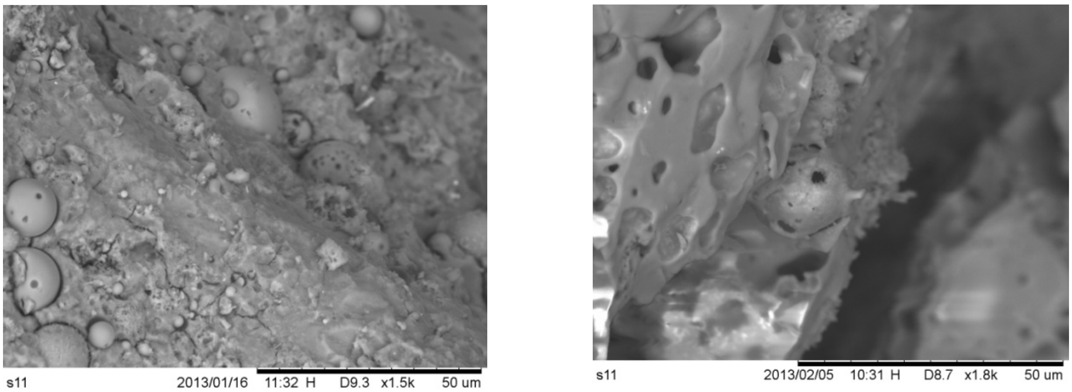
1. The loss of free water and chemically bound water in the $CaOH_2$ through dehydroxylation leads to significant dehydration of the concrete and loss of chemical bonding and induces spalling [12].

2. The disparity in the amount of thermal expansion in the aggregate in comparison to the shrinkage of the OPC cement means that micro stresses are induced and leads to cracking in the concrete.

3. Different types of aggregate have a significant effect on the strength-temperature relationship of concrete. Calcareous aggregates decarbonise at temperatures above 800°C. This can be avoided by using siliceous aggregates [11]. Yet quartz also undergoes a phase change at around 400^{o} C (configuration α to β) , which results in very high thermal expansion.

4. The rate of heating has little effect on the sample so long as the temperature gradient is below 10 degrees per centimetre towards the core [11].

In previous research focusing on the residual properties of geopolymer paste and mortar, it has been found that fly ash based geopolymers have retained most of its residual strength after being exposed to an elevated temperatures [12-18]. This is attributed to the fly ash based geopolymer having large numbers of small pores that act as a conduit for water as it escapes the structure and probably more importantly to the fact that GPC does not use a hydraulic binder. This means that there is minimal spalling and damage to the geopolymer structure. The high strength retention can also be due to the gradual sintering of the un-reacted fly ash that is present within the concrete [15-17]. Sintering occurs when very fine powders (such as fly ash) are elevated to high temperatures causing molecules to diffuse to the boundaries of the particles, which causes the particles to partially fuse together. This means that the unused fly ash powder, which is responsible for local weaknesses before exposure, bonds covalently into a strong solid material within the concrete (Figure 1).



a) Before Exposure *b) After Exposure to 1000°C*

Figure 1: SEM Images of GPC (a) before and (b) after temperature exposure.

MATERIAL AND METHODS

Since extensive research is available on the performance of OPC at elevated temperatures [10, 11, 19-25], the current research focuses on comparing the residual properties of GPC with normal and FLASHAG aggregates. Grade D Sodium Silicate and 12M Sodium Hydroxide prepared from 98% pure flakes were used as alkaline solutions while ASTM Class F Fly Ash was used as the main aluminium and silicate source. Cylinders with nominal measurements of 75mm diameter and 150mm height were mixed and cast using the procedure described in [26]. The samples, after an initial rest period, were cured for 72 hours at 80°C. They were then placed in an environmental room with a relative humidity of 50% and a temperature of 23°C till the time of testing. Samples containing normal aggregates are identified as M13 while those containing FLASHAG are identified as M4Sp. The composition and the ambient mean compressive strength (f_c') is given in Table 1.

Table 1: Composition and Mean Compressive Strength (MCS) of the GPC Samples

	FA (kg/m3)	NaOH (kg/m3)	Na2SiO3 (kg/m3)	Coarse (kg/m3)	Fine (kg/m3)	Water (kg/m3)	SP (kg/m3)	f_c' (MPa)
M13	420	44	110	1130	570	56.2	8	38
M4Sp	440	65	162.5	660	340	11	8	40

The samples were heated at 4.5°C/min until they reach the target temperature, the target temperature is then maintained for 2 hours to ensure that thermal equilibrium is achieved. The furnace is then turned off and the samples are left in the chamber to cool down to ambient temperature. The samples are then removed from the chamber, and tested for compressive strength ($f_c^{\prime}$), modulus of elasticity (E_c) and splitting tensile strength (f_{ct}). Samples are also weighed before and immediately after cooling down to record the change in weight of each sample. Strain measurements for the compressive strength and modulus tests are done using a laser extensometer and reflective strips on the samples. Data is acquired using a custom *Virtual Insturment* on LabView™. Figure 2 shows the illustrative test setup for compressive and modulus tests while no strains were measured during the indirect tensile tests. Loading rates for all tests comply with the AS3600 standards.

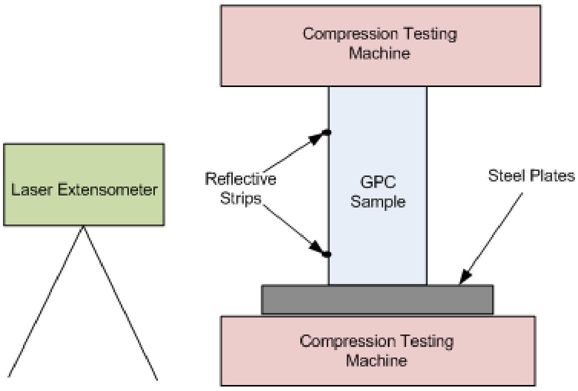


Figure 2: Test Setup

RESULT AND DISCUSSION

The stress-strain curves for geopolymer concrete with and without FLASHAG aggregates for different temperatures are given in Figure 3 (a and b). It is worth noting that specimens from both mixes at all temperatures failed soon after reaching their peak strength, displaying the brittle nature of GPC. However, the mix containing FLASHAG (M4-SP) develops slightly higher strains at failure at ambient temperatures when compared to GPC containing normal aggregates. No significant reduction in stiffness is observed for M13 samples when heated to 200°C, although there is a marked reduction in the stiffness for temperatures of 400°C and over. In fact the modulus of elasticity is reduced by 60% at 800°C and by over 80% when the exposure temperature is 1000°C. A similar pattern is observed for the M13 samples in compression and splitting (Figure 4 a, b and c). After a slight increase in both compression and splitting values at 200°C, due to further geopolymerization, there is a steep decline in both properties as the temperature is raised.

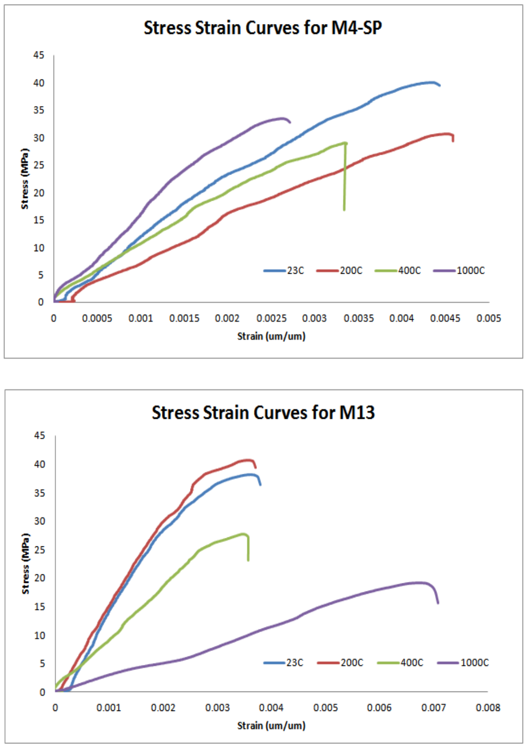
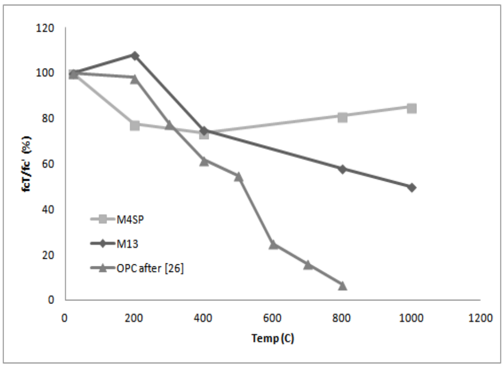
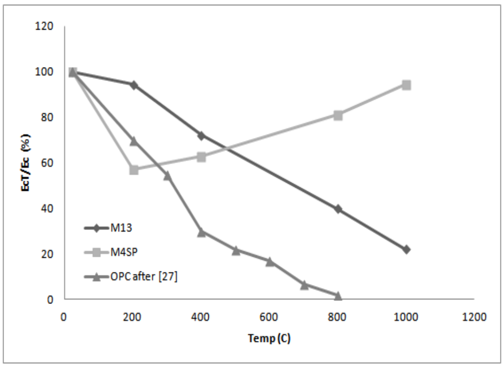


Figure 3: Stress Strain Relationship for the GPC mixes

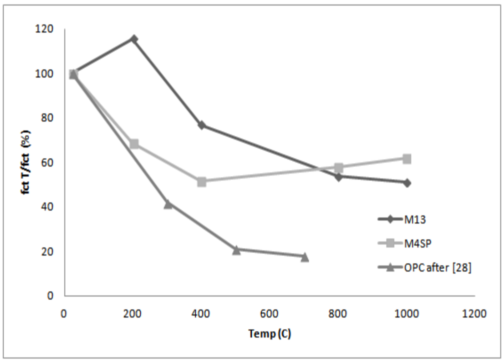
One possible explanation for this change in behaviour may be the 'glass transition' of GP matrix which is characterised by abrupt loss of stiffness and strength. Although this occurs at a temperature of 520°C in GP pastes [18], the presence of aggregates and use of varying source material may affect the exact value of this temperature. It is also observed that normal GPC retains almost 50% of splitting and compressive strength after 1000°C exposure. This is much higher than OPC, which retains about 5% of its compressive strength and less than 20% of its splitting strength at 800°C. The loss of stiffness in OPC is more accentuated as the temperature is increased over 200°C [27-29]. A rather unique behaviour is observed for M4-SP samples. At temperatures of 400°C and under the samples steadily lose their stiffness and strength. Since FLASHAG are lightweight aggregates with numerous voids, they carry a significant amount of water to attain saturated surface dry (SSD) condition. During heating this free water escapes leaving behind voids in the matrix. However the temperature is still not high enough to bring about a change in the material behaviour. These voids left behind by the escaping water contribute to the loss of stiffness and strength at relatively low temperatures. Nevertheless at higher temperatures M4-SP samples exhibit far superior strength and stiffness properties. At 800°C the samples regain all but 20% of their compressive strength and modulus of elasticity, while the splitting strength is also increased from 50% at 400°C to over 60% at 800°C of ambient values. This trend continues as the temperate reaches 1000°C. The residual values of compressive strength, modulus of elasticity and splitting strength after exposure to 1000°C was recorded as 85%, 95% and 62% of ambient, respectively. One important reason why the strength of FLASHAG-OPC concrete surpassed normal aggregate-OPC concrete is the tight aggregate-matrix bond accomplished with FLASHAG [5]. This characteristic is even more accentuated in the case of FLASHAG-GPC where the matrix is essentially compatible with the aggregates. Thus a plausible explanation for this observation may be that as the temperature is increased over and above 400°C, the fly ash particles present in both the matrix and the aggregates, fuse together to form a near-monolithic phase as shown on Figure 5b. Moreover, the



a) Compressive Strength as Function of T



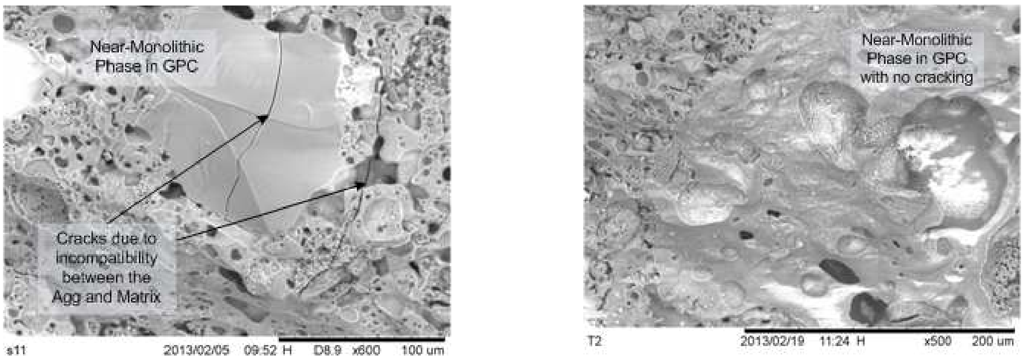
b) Modulus of Elasticity as Function of T



c) Splitting Strength as Function of T

Figure 4: Variation in GPC Properties as a Function of Temperature

presence of fused particles throughout the matrix may contribute towards the increase in strength and stiffness of M4-SP samples after 400^{o}C. Obviously more study is required and is actually underway to better understand this phenomenon.



a) View of GPC Matrix containing Normal Aggregates (1000^{o}C)

b) View of GPC Matrix containing FLASHAG Aggregates (1000^{o}C)

Figure 5: SEM Images of GPC with a) Normal Aggregates and b) FLASHAG Aggregates

While similar geopolymer matrix is present in both M13 and M4-SP, yet during the cooling period, cracks are induced in the normal GPC matrix due to incompatibilities between the GP matrix and the normal aggregates. The FLASHAG, however, contains numerous voids and thus acts more or less like tiny springs distributed throughout the samples, reducing the stresses developed due to cooling and hence results in reduced cracking. This hypothesis is supported by the SEM study of the GP matrix containing the two different aggregates (Figure 5).

CONCLUSIONS

A detailed study was conducted to research the behaviour of GPC with normal and special aggregates after exposure to extreme temperatures and to compare the results with published results of OPC. It was found that when exposed to extreme temperatures GPC, both with normal and special aggregates, outperforms OPC. Reported data indicate that OPC loses substantial structural integrity at temperatures over 400^{o}C. GPC on the other hand, retain over 60% of its strength and stiffness at similar temperatures. At even higher temperatures, GPC containing aggregates, which were manufactured from fly ash as their basic component, has outmatched the performance of normal aggregate GPC with properties almost equivalent to those at ambient temperatures.

REFERENCES

[1] *J. Davidovits, "Soft Mineralogy and Geopolymers," in Proceedings of the Geopoolymer 88 International Conference, France, 1988.*

[2] *J. Davidovits, "Geopolymers Inorganic polymerie new materials," Journal ofThamalAnalysis, vol. 37, pp. 1633-1656, 1991.*

[3] *N. A. Lloyd and B. V. Rangan, "Geopolymer Concrete with Fly Ash," presented at the Second International Conference on Sustainable Construction Materials and Technologies, Italy, 2010.*

[4] *A. Fernandez-Jimenez and A. Palomo, "Alkaline Activation of Fly Ashes. Manufacture of Concrete Not Containing Portland Cement," presented at the International RILEM Conference on the Use of Recycled*

Materials in Buildings and Structures, 2004.

[5] *O. Kayali, "Fly ash lightweight aggregates in high performance concrete," Construction and Building Materials, vol. 22, pp. 2393-2399, 2008.*

[6] *I. Diaz-Loya, et al., "Mechanical Properties of Fly-Ash-Based Geopolymer Concrete," ACI Materials Journal, vol. 108, pp. 300-308, 2011.*

[7] *T. J. Gourley and G. B. Johnson, "Development in Geopolymer Precast Concrete," in International Workshop on Geopolymers and Geoplymer Concrete, Perth, Australia, 2005.*

[8] *W. D. A. Rickard, et al., "Assessing the suitability of three Australian fly ashes as an aluminosilicate source for geopolymers in high temperature applications," Materials Science and Engineering A, vol. 528, pp. 3390-3397, 2011.*

[9] *I. Diaz-Loya, et al., "Factors Affecting the Suitability of Fly Ash as Source Material for Geopolymers," Fuel, vol. 89, pp. 992-996, 2010.*

[10] *A. Neville, Properties of Concrete, 4th ed.: Longman Group Limited, 1995.*

[11] *U. Schneider, "Concrete at high temperatures—a general review," Fire Safely Journal, vol. 13, pp. 55-68, 1988.*

[12] *M. Guerrieri, et al., "Residual strength properties of sodium silicate alkali activated slag paste exposed to elevated temperatures," Materials and Structures, vol. 43, pp. 765–773, 2010.*

[13] *A. Elimbi, et al., "Effects of calcination temperature of kaolinite clays on the properties of geopolymer cements," Construction and Building Materials, vol. 25, pp. 2805–2812, 2011.*

[14] *J. G. S. v. Jaarsveld, et al., "The effect of composition and temperature on the properties of fly ash- and kaolinite-based geopolymers," Chemical Engineering Journal, vol. 89, pp. 63-73, 2002.*

[15] *D. L. Y. Kong and J. G. Sanjayan, "Damage behavior of geopolymer composites exposed to elevated temperatures," Cement & Concrete Composites, vol. 30, pp. 986–991, 2008.*

[16] *D. L. Y. Kong and J. G. Sanjayan, "Effect of elevated temperatures on geopolymer paste, mortar and concrete," Cement and Concrete Research, vol. 40, pp. 334-339, 2009.*

[17] *D. L. Y. Kong, et al., "Comparative performance of geopolymers made with metakaolin and fly ash after exposure to elevated temperatures," Cement and Concrete Research, vol. 37, pp. 1583-1589, 2007.*

[18] *Z. Pan and J. G. Sanjayan, "Stress–strain behaviour and abrupt loss of stiffness of geopolymer at elevated temperatures," Cement & Concrete Composites, vol. 32, pp. 657–664, 2010.*

[19] *Z. P. Bazant and M. F. Kaplan, Concrete at high temperatures. Material Properties and Mathematical Models: Longman House, Burnt Mill, 1996.*

[20] *W. Khaliq and V. Kodur, "Thermal and mechanical properties of fiber reinforced high performance self-consolidating concrete at elevated temperatures," Cement & Concrete Research, vol. 41, pp. 1112–1122, 2011.*

[21] *A. Khennane, "Mechanical modelling of the behaviour of concrete at elevated temperatures," PhD, Department of Civil Engineering, The University of Queensland, Australia, 1992.*

[22] *A. Khennane and G. Baker, "Uniaxial model for concrete under variable temperature and stress," Journal of Engineering Mechanics, ASCE, vol. 119, pp. 1507-1525, 1993.*

[23] *G. A. Khoury, et al., "Transient thermal Strain of concrete: literature review, conditions within specimen and behaviour of individual constituents," Magazine of Concrete Research, vol. 37, pp. 131-144, 1985.*

[24] *L.-y. Li and J. Purkiss, "Stress–strain constitutive equations of concrete material at elevated temperatures," Fire Safely Journal, vol. 40, pp. 669–686, 2005.*

[25] *A. N. Noumowe´, et al., "Thermal stresses and water vapour pressure of high performance concrete at high temperature," in Fourth International Symposium on the Utilisation of High Strength/High Performance Concrete, Paris, France, 1996, pp. 561-570.*

[26] *M. J. Talha, et al., "Mix Design Procedure for Alkali Activated Fly Ash-based Geopolymer Concretes," in International Conference on Engineering and Applied Science, Beijing, China, 2012, pp. 139-152.*

[27] *Z. Lu, "A Research on fire response of reinforced concrete beams," PhD, Civil Engineering, Tongji University, 1989.*

[28] *W. Tan, "A research on reinforced concrete beams subjected to high temperatures and expert system," Civil Engineering, Tongji University, 1990.*

[29] *Q. Z. Xie GM, "Research on bond and tension behaviour of concrete after high temperature," Journal of Zhejiang University, vol. 32, pp. 597–602, 1998.*