Qualitative and quantitative analysis and identification of flaws in the microstructure of fly ash and metakaolin blended high performance concrete after exposure to elevated temperatures

Abid Nadeem [a], Shazim Ali Memon [b,*], Tommy Yiu Lo [b]

[a] Department of Civil and Architectural Engineering, Qatar University, Doha, Qatar
[b] Department of Civil and Architectural Engineering, City University of Hong Kong, Hong Kong

HIGHLIGHTS

▶ Qualitative and quantitative analysis of HPC blends at elevated temperature was carried out.
▶ Three types of flaws were identified namely texture flaws, orientation flaws and local flaws.
▶ Changes in the structure of ITZ were classified into three temperature regimes.

ARTICLE INFO

ABSTRACT

The extensive use of eco-friendly materials in concrete has led to the demand to fully understand the effect of fire on concrete. This research was carried out to evaluate high performance concrete (HPC) made with fly ash and metakaolin with replacement level of 20 percent by weight of cement after elevated temperatures exposure (200 °C, 400 °C, 600 °C and 800 °C). The mechanical performance was assessed from compressive strength while the durability was assessed from chloride permeability and water sorptivity test. Qualitative analysis of the microstructure of heated and unheated concrete was performed by SEM while quantitative analysis was performed on SEM images using Image Pro-plus software. Based on the qualitative and quantitative analysis of SEM images, the distribution of the number, type and surface area fraction of flaws were identified and changes in the structure of Interfacial Transition Zone (ITZ) were classified into different temperature ranges.

Test results show that for all mixes post-elevated temperature compressive strength decreased while charge passed and sorptivity values increased with the increase in temperature from 27 °C to 800 °C. For all mixes, major strength and durability loss occurred after 400 °C. Therefore, 400 °C can be considered as critical temperature from the standpoint of strength and durability loss. From the qualitative and quantitative analysis, different types of flaws were identified. These were texture flaws (T), orientation flaws (O) collectively called as textured and orientation flaws (TO) and local flaws (L). The post-elevated temperature surface area fraction of TO and local flaws in the ITZ of each concrete mix continuously increased with the increase of elevated temperature. The increase in surface area fraction of flaws resulted in gradual loss in the mechanical and durability properties of concrete. Major increase in surface area fraction occurred in between 400 and 600 °C, resulting in major strength loss and sharp increase in charged passed and sorptivity values through concrete specimens. Therefore, 400 °C can be regarded as critical for change in the properties of concrete. No specific relationship between the number of TO flaws and the type of binding material in concrete and the temperature was found. However for local flaws, in general, the number increased with the increase of temperature. Also, the changes in the structure of ITZ were classified into three temperature ranges namely; the low range temperatures (27–200 °C), the medium range temperatures (200–400 °C) and the high range temperatures (400–800 °C). The physical character of ITZ in HPC changes gradually from a discrete or discontinuous flaw zone at normal or mildly elevated temperature to a continuous and highly porous flaw zone at elevated temperatures. Thus, the classification signifies the effect of the texture of coarse aggregate and the orientation of fine aggregate in concrete matrix coupled with the effect of elevated temperatures on ITZ of HPC.

© 2012 Elsevier Ltd. All rights reserved.

* Corresponding author. Tel.: +852 62090030.
  E-mail addresses: abid@qu.edu.qa (A. Nadeem), shazimalimemon@gmail.com (S.A. Memon).

1. Introduction

Concrete is the most widely used construction material. Man consumes no material except water in such tremendous quantities [1]. The extensive use of concrete as a structural material has led to the demand to fully understand the effect of fire on concrete. The issues related to the exposure of concrete to the elevated temperatures pose concerns about the serviceability and stability of the structure which, in turn, are related to human safety.

The inclusion of eco-friendly materials to improve the mechanical and durability properties of HPC is increasing day by day due to their superior structural performance, environmental friendliness and energy conservation implications. This in turn requires the assessment of its fire resistance capabilities [2]. Many researchers have assessed the mechanical and durability performance of fly ash concrete after exposure to elevated temperatures. It was found that the fly ash improved the performance of concrete at elevated temperatures and this improvement was more significant at temperatures below 600 °C [3]. Poon et al. carried out investigation into post-elevated temperature performance of concrete made with metakaolin [4]. They found that concrete with higher replacement of metakaolin (20%) suffered higher loss in strength at all temperatures and suggested that severe durability loss occur at elevated temperatures. These and many other researches [5,6] used mercury intrusion porosimetry as a tool to investigate the connection between the loss in compressive strength and durability while the current research uses Image Analysis technique to scientifically determine the degradation in mechanical and durability properties of HPC made with metakaolin and fly ash.

The post-fire residual properties of concrete stems from considerable modifications undergone by its components, namely the physicochemical changes in the cement paste and in the aggregates, coupled with the thermal incompatibility between them [7]. At low temperatures i.e. 70–80 °C ettringite dissociates while at around 100 °C water physically bound in both cement matrix and the aggregates starts evaporating causing minor loss of strength [8]. At 300 °C, the evaporation of inter layer water from calcium silicate hydrate structure and combined water from calcium silicate hydrate and sulfoaluminate hydrates occur [9]. In between 400 and 600 °C, series of reaction may be activated resulting in decomposition of hydration products and the destruction of calcium silicate hydrate gels [10]. Decomposition of calcium hydroxide takes place above 350 °C. This may lead to severe damage to strength due to lime expansion during the cooling period [11]. The transformation of quartz from α to β form at around 573 °C may also cause distress in concrete [12].

ITZ is widely regarded as the third component of concrete microstructure in addition to cement paste and aggregate. Consideration of ITZ is important from the point of view that it has been observed to be a weak link between paste and aggregate. Thus, the strength and other properties of concrete might be controlled by the properties of ITZ. This understanding of ITZ was gradually recognized since mid 1980s [13]. Scanning electron microscope (SEM) studies have helped to discover this third component of concrete which was characterized by more porous and heterogeneous structure than bulk matrix.

Two reasons are normally given for ITZ being more porous than rest of the concrete matrix. It is stated that a thin water film at the surface of aggregate is created for a thickness of about 10 μm [14]. Cement grains are not present within this water film. Second view for porous ITZ is the wall effect in which cement grains are homogenously packed in unrestricted spaces but due to the presence of aggregate, there is a restriction for the homogenous distribution of cement grains [15]. The aggregate particle acts as a wall to disperse cement grains away from the aggregate. Based on the studies performed on ITZ, researchers have tried to characterize the ITZ

into models. Two such models are normally cited. The first model describes ITZ as layered structure where aggregate is covered first by a thin 'duplex film' of calcium hydroxide (CH) and calcium silicate hydrate (CSH) then a transition layer of high porosity and then bulk cement paste. This model was developed for neat cement paste cast against glass [16] and rock aggregate [17]. A variation in this model also exists. This modification did not propose duplex film; rather ITZ was a combination of CH, CSH and pores [1]. As, these models of ITZ were from laboratory made concretes and under simplified conditions concerning the arrangement of aggregates, they were not very adequate in describing the properties of real concrete. Real concrete is more irregular than the concrete produced in laboratory. The nature of porosity in mortar paste in concrete is different from the one in neat cement paste cast against a flat polished aggregate [18].

Many studies have been performed in the past using flat and polished surface of aggregate. However, it could promote the formation of water film at the aggregate surface which may not correspond to real concrete situation [19]. Real concrete often includes aggregate particles which are not smooth. Therefore, there is a need to characterize ITZ taking into consideration the effect of rough aggregate particles. The study in this paper thus examines the microstructure of ITZ in HPC made with crushed granite aggregate particles after exposure to normal and elevated temperatures by the analysis of images obtained from SEM. SEM was used in secondary electron mode which has been found more useful for fractured specimens [20]. Based on the qualitative and quantitative analysis of SEM images, the distribution of the number, type and surface area fraction of flaws were identified and changes in the structure of ITZ were classified into different temperature ranges.

2. Experimental investigation

2.1. Cementitious materials

Ordinary Portland cement complying with BS12: 1991, low-calcium pulverised fuel ash complying with BS3982: Part 2: 1982 and metakaolin were used as cementitious materials. The chemical composition and physical properties of these materials are given in Table 1.

2.2. Aggregates

The coarse and fine aggregates employed were crushed granite and river sand complying with the requirements of BS 882: 1992. Coarse aggregates of two sizes 20 mm and 10 mm were used. The particles of coarse aggregates had the specific gravity of 2.62 and 24 h absorption value of 0.6% while fine aggregate particles had specific gravity of 2.61, 24 h water absorption 0.7% and fineness modulus of 2.4.

Table 1
Composition of OPC, FA and MK.

Chemical composition (%)	OPC	FA	MK
Silicon dioxide (SiO_2)	19.6	56.8	53.2
Aluminum oxide (Al_2O_3)	7.3	28.2	43.9
Ferric oxide (Fe_2O_3)	3.3	5.3	0.38
Calcium oxide (CaO)	63.1	3.0	0.02
Magnesium oxide (MgO)	2.5	5.2	0.05
Sodium oxide (Na_2O)	0.1	–	0.17
Potassium oxide (K_2O)	1.1	–	0.10
Sulfur trioxide (SO_3)	2.1	0.7	–
Loss on ignition	3.0	3.9	–
Physical properties			
Specific gravity	3.16	2.31	2.62
Specific surface (m^2/kg)	312	412	12680
Compressive strength 3 days (MPa)	35		
7 days	45		
28 days	60		
Initial setting time (min)	125		
Final setting time (min)	240		