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## Impact of high temperature on PFA concrete

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#### Abstract

After being subjected to high temperatures, the residual properties of pulverized fly ash (PFA) concrete have been investigated. Both mechanical and durability properties of concrete were tested on concretes made with different water to binder ratios and PFA contents. Microscopic techniques were then employed and the pore structure and microhardness values of hardened cement paste (hcp) were determined. The results of rapid chloride diffusion tests revealed that concrete durability deterioration commences after exposure to temperatures which are lower than those at which compressive strength deterioration commences. The rise in compressive strength, which occurs after exposure to 250°C, may be largely due to the hardening of cement paste caused by drying and the further hydration of cementitious materials. The simultaneous loss of durability, however, can be explained by a weakened transition zone between hcp and aggregate, and by the concurrent coarsening of the hcp pore structure. When PFA is included, an improvement of fire resistance as characterized by the residual compressive strength was observed, and this relative improvement over non-PFA concrete was the most pronounced for maximum exposure temperatures of 450°C and 650°C. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Compressive strength; Diffusion; Microstructure; Fly ash; High temperature

## 1. Introduction

Fire remains one of the main menaces to modern buildings. With the current extensive use of pulverized fly ash (PFA) in structural concrete and the rise in fire occurrences in recent years, a thorough understanding of the impact of fire on PFA concrete is urgently needed. As concrete is a non-combustible material, the effects of fire on the properties of concrete are the effects caused by exposure to elevated temperatures and much of the past research has been carried out on concrete exposed to high temperatures as opposed to exposure to real fires. Since the 1920s [1], many investigations have been carried out on the residual mechanical properties of concrete subjected to elevated temperatures, but limited data on the deterioration of durability of PFA concrete are available from previous work. Limited conclusions only, therefore, have been drawn for PFA concretes, where damage is not only due to a combination of chemical and mechanical changes to the constituents of concrete, but is greatly influenced by

factors such as the properties of the PFA itself, the dosage of PFA, and the curing regime.

Nasser and Marzouk [2] conducted a series of tests on mass concrete containing fly ash exposed to high temperatures ranging from 21.4°C to 323°C. Tests were carried out on concrete made with a 25% replacement of cement by weight and a w/b ratio of 0.6. The exposure time lasted up to over 6 months. They found that there was an increase in strength of concrete in the temperature range 121°C to 149°C, and in one case the increment was as high as 52% of the unheated value. However, the strength and elasticity of concrete were simultaneously reduced to around one half and one quarter after exposure to temperatures 177°C and 232°C, respectively. They then explained that the increase of strength was due to the formation of tobermorite, and the deterioration was due to the transformation of tobermorite (gel) into poorer binding materials.

The effects of high temperature and high pressure on high strength concrete incorporating both silica fume and fly ash were also investigated by Ghosh and Nasser in 1996 [3]. The fly ash content was 20%, or 60% of the weight of binder while silica fume was used in each mix with a constant dosage of 10%. The 28 days strengths of the two mixes of concrete were 72.1 and 54.1 MPa.

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They found that the compressive strength and the static modulus decreased gradually after specimens were subjected to temperatures ranging from 21.4°C to 232°C and high pressures from 5.2 to 13.8 MPa. Microstructural analysis revealed that the deterioration was due to the physicochemical transformation of the gel structure. They declared that the transformation of the matrix seemed to consist of purely physical changes at exposure temperatures lower than 71°C, and the chemical transformation of gel at higher temperatures up to 232°C might be the main cause of the reduction in strength and elastic modulus.

Since concrete can be deemed as a two-phase material formed by aggregate embedded in hardened cement paste (hcp), the properties of cement paste play a very important role in the behavior of concrete. Normally, aggregates are more stable than hcp when exposed to high temperatures, although decomposition of some less thermally stable aggregate may occur when exposure temperatures are extremely high [4]. Knowledge of the effects on hcp of exposure to elevated temperatures is thus necessary for the better understanding of the residual properties of concrete.

Grainger [5] carried out some research on cement/fly ash pastes subjected to a series of temperatures ranging from 100°C to 600°C with an interval of 100°C. In his research, the dosages of PFA were 20%, 25%, 37.5%, and 50% of the total weight of binders. It was found that the addition of PFA could improve the residual compressive strength of cement paste, and the improvement was especially significant for exposure temperatures above 300°C. He also observed an increase of strength in all specimens exposed to temperatures from 150°C to 300°C. And for those specimens containing lower contents of PFA, the increases in strength occurred at lower temperatures. However, Sarshar and Khoury [6] reported that compressive strengths of cement and cement/PFA pastes decreased after being heated to temperatures ranging from 100°C to 600°C at a rate of 1°C/min. The beneficial effect of the presence of PFA, however, was in good agreement with the results of Grainger for exposure temperatures above 300°C.

Severe cracking of the heated concrete during postcooling has been observed by many researchers since the 1920s [7–9]. The severe cracking can be ascribed to the rehydration of dissociated Ca(OH)<sub>2</sub> resulting in a 44% volume increase [9]. Therefore, the reduced calcium hydroxide in cement paste containing PFA, which is due to pozzolanic reaction, may lead to reduced cracking. Dias et al. [10] conducted tests on cement pastes containing 10%, 25%, and 40% PFA replacement by weight and found that even a 10% fly ash replacement can eliminate all visible surface cracking in specimens during postcooling after exposure to 600°C. They also pointed out that 400°C was a critical temperature for portland cement concretes, above which concretes would disintegrate on subsequent postcooling exposure to ambient conditions.

## 2. Research significance

Ever since PFA concretes at high temperatures were investigated in the 1960s, it has been recognized that PFA concrete suffers less deterioration in its mechanical properties than concrete made with ordinary portland cement (OPC) only. However, a further probe into the change in the microstructure of PFA concrete subjected to high temperatures is still lacking, although some research has been carried out on the pore structure of concrete [11,12]. Also lacking is knowledge on the reduction in durability of PFA concrete subjected to high temperatures.

During the research described in this paper, concretes made with PFA replacements of 0, 25% and 55% were investigated. The w/b ratios were 0.5 and 0.3, making concretes representative of normal strength and high strength, respectively. Residual compressive strengths were determined on PFA concrete subjected to high temperatures ranging from 250°C to 800°C. Deterioration of the durability of PFA concretes was quantified by use of the rapid chloride diffusion test according to ASTM C1202 [13]. To obtain an insight into the residual properties, the pore size distribution was determined by the mercury intrusion porosimetry (MIP) technique, and the microhardness of hcp within the transition zone between aggregate and paste was measured. The results of the microscopic investigations gave a good explanation of the change in macro behavior of PFA concretes in comparison to concretes made with OPC only.

## 3. Experimental details

## 3.1. Materials

The binders used in this research are locally available OPC complying with ASTM Type I and low-calcium fly ash equivalent to ASTM class F. Their chemical compositions and physical properties are given in Table 1.

A superplasticizer named "Super 20" was adopted for fabricating high strength concrete with a w/c ratio of 0.3. It is a naphthalene-based superplasticizer available as a dark brown liquid containing 38.6% solid.

The fine aggregates and coarse aggregates are local natural river sands and crushed granite, respectively. Coarse aggregates of 10 and 20 mm were mixed in a ratio of 2:3.

Table 1 Chemical composition and physical properties of binders

	Chemical composition (%)						Physical properties		
Binder	$SiO_2$	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	$SO_3$	Loss on ignition	Specific gravity	Specific surface (m²/kg)
		7.33 28.21		63.15 <3.00				3.16 2.31	3519.5 4120

## 3.2. Mix proportions

The mix proportions are shown in Table 2.

## 3.3. Sample preparation

Several 100 mm cubes were cast for determining the compressive strength of concrete. Samples obtained from cores taken from crushed cubes after the compression tests were soaked in acetone to stop the hydration for MIP tests, and then they were dried at 60°C for 24 h before measurement. Special precautions were taken to make sure that no coarse aggregates were included in the samples. Several 100 mm diameter × 200 mm height cylinders were cast for rapid chloride diffusion tests according to ASTM C1202 [13] to investigate the influence of high temperature exposure on the durability of concrete. To eliminate the influence of dispersed sands on the transition zone, mixes without fine aggregates were also cast into cylinders. Slices cut from the middle of the cylinders were polished with 600# paper then 1500# paper to obtain an adequate surface with a minimum of damage. Slices were then carefully sealed to avoid carbonation, which might lead to larger measured hardness values because the carbonation product (CaCO<sub>3</sub>) is much harder than the reactants.

## 3.4. Curing regime

Specimens were demolded 24 h after casting, and cured in a water tank kept at 25°C until the age of 7 days. Thereafter all specimens were moved into a curing chamber with a controlled temperature of 25°C and 75% relative humidity (RH). Specimens were heated to targeted temperatures at the age of 90 days, and their residual properties were then investigated.

## 3.5. Heating regime

In order to obtain evenly heated specimens, specimens were heated at a slowly increasing rate of 1°C/min in an electric oven. When the targeted peak temperature was reached, the oven temperature was maintained for 60 min. The oven was then switched off, the oven doors opened

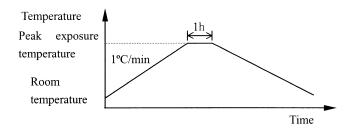


Fig. 1. Temperature rising regime.

and the specimen was left to cool over many hours until room temperature was reached gradually. This is illustrated by Fig. 1.

## 3.6. Testing procedure and methods

Compressive strengths of cubes were measured according to BS 1881: Part 116:1983 [14]. Chloride diffusion tests were conducted on  $51\pm1$  mm thick slices, which were sawn from cylindrical specimens followed by vacuum saturation treatment, complying with the prescription by ASTM C1202-97 [13]. The reported data are average values from three companion specimens.

Samples for determining the pore size distribution of hcp were taken from the cores of the crushed cubes. In this research the measured pore sizes ranged from  $10^{-8}$  to  $10^{-4}$  m, while the pressure ranged from 210 kPa to 210 MPa. The contact angle was assumed to be  $140^{\circ}$  and the mercury surface tension was taken as 0.485 N/m.

Based on the ASTM E384-99 [15] testing method, a Vickers indenter was used in this research to determine the microhardness in bulk pastes and in the transition zone between aggregates and hcp. As the typical width of the interfacial transition zone (ITZ) is about 50  $\mu m$ , the applied load in the microhardness test was determined to be 0.049 N so that the spacing between indentation points should be at least two times the diagonal of the indentation while ensuring a sufficient number of points was taken to map the ITZ [16]. The strategy for selecting the indentation points for the measurements is presented in Fig. 2. The measured range was up to 250  $\mu m$  away from the surface of aggregates. Six determinations were performed on the surface of each

Table 2 Mix proportions (per cubic meter)

		Replacement				Coarse		Superplasticizer
Mix	w/b	ratio (%)	OPC (kg)	PFA (kg)	Sand (kg)	aggregate (kg)	Water (kg)	(L)
C-0.5-00	0.5	0	410	0	609	1132	205	0
C-0.5-25	0.5	25	307.5	102.5	576	1132	205	0
C-0.5-55	0.5	55	184.5	225.5	536	1132	205	0
C-0.3-00	0.3	0	500	0	724	1086	150	10.5
C-0.3-25	0.3	25	375	125	683	1086	150	10.5
C-0.3-55	0.3	55	225	275	634	1086	150	10.5

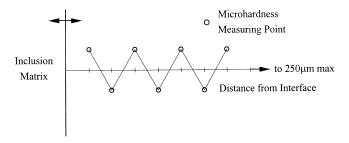


Fig. 2. Strategy for selecting indentation points.

sample, and the profiles of microhardness yielded by the average values derived.

### 4. Test results and discussions

## 4.1. Discussions on macro properties: compressive strength and chloride diffusion

Normally compressive strength is the main concern when assessing the mechanical strength of concrete. After exposure to 250°C, the test results given in Fig. 3 indicate that concretes showed no reduction in compressive strength but, rather, a gain of 8-15% over the basic strength when tested without any prior heating. For concrete made with OPC only, the increment was 8-9%, while PFA concrete gained about 10–15%. After exposure to a higher temperature of 450°C, high volume PFA concrete exhibited a much better fire resistance than other mixes from the viewpoint of strength loss. Only a negligible proportion of the original strength was lost for the C-0.5-55 and the C-0.3-55 mixes, the loss being not more than 4%. The strength losses for concretes made with 25% PFA and without PFA differed between 18-14%. Even for an exposure temperature of 650°C, the beneficial effect of the high PFA dosage was still significant, resulting in a residual compressive strength of 65.8% of the original unheated strength for high PFA content concretes, while only 51.1–56.2% of the original strength was maintained when the other four mixes were exposed to the same temperature. The effect of the addition of PFA diminished when the exposure temperature was raised to 800°C.

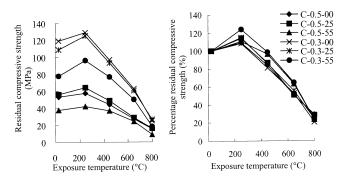


Fig. 3. Residual compressive strength and percentage to original value.

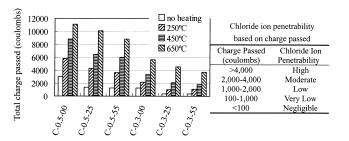


Fig. 4. Total charge passed in chloride diffusion tests.

PFA concretes are generally thought to be more durable because they have good resistance to chloride penetration under normal conditions [17,18]. However, as is shown in Fig. 4, when PFA concrete and conventional concrete were exposed to a temperature of 250°C, severe deterioration in permeability was observed. Based on a rapid chloride diffusion test method prescribed by ASTM C1202-97 [13], the total charges passed after exposure to 250°C became two to four times those for the unheated concrete. As previously discussed, concrete developed a higher strength after exposure to this temperature. These results infer that more attention should be paid to the permeability of concretes when they are exposed to a medium temperature of about 250°C. Using the qualitative terms (see Fig. 4) of ASTM C1202, C-0.5-00 degraded from "moderate" to "high" chloride ion penetrability, C-0.5-25, C-0.3-00, and C-0.5-55 degraded from "low" to "moderate" or "high," while C-0.3-25 and C-0.6-55 from "very low" to "low." Concrete made with a PFA dosage of 25% suffered a greater loss in resistance to chloride penetration than plain OPC concretes did. After exposure to 450°C and higher temperatures, concrete with a w/b ratio of 0.5 degraded to "high," and concrete with a w/b ratio of 0.3 could still be rated as "moderate." For the exposure temperature of 650°C, all concretes except C-0.3-55 degraded to "high" chloride ion penetrability. The relative charges passed, expressed as percentage of total charges passed after heating to the original values of the unheated specimens, are shown in Fig. 5. The figure indicates PFA concretes suffered more damage than OPC concretes in terms of chloride penetrability after exposure to high temperatures.

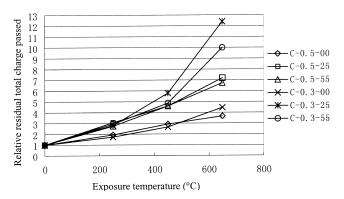


Fig. 5. Relative residual total charge passed as percentage to original values.

# 4.2. Discussions on microscopic test results: microhardness and pore structure in hcp

Microhardness testing has been introduced as a powerful tool for characterizing the microstructural properties of hcp and ITZ [19,20]. In this research, this testing method was applied to characterize the influence of high temperature on the properties of bulk paste and the ITZ around aggregates. When the exposure temperatures were higher than 450°C, cracking along the boundary between aggregate and hcp was inevitably obvious, making it impossible to obtain accurate data on the ITZ. Therefore, the reported microhardness values in the ITZ in this research are results on unheated concretes and concretes exposed only to 250°C. Also reported are microhardness test data on bulk pastes in unheated concretes and in concretes exposed to 250°C, 450°C, and 650°C.

As also shown in Fig. 6, increased ITZ ranges, i.e., where the microhardness test resulted in lower Vickers Hardness Numbers (Hv) than those in the middle of the hardened bulk cement pastes, were observed after concretes were exposed to 250°C. For concretes made with OPC only, the extent of the weak zones increased roughly by 40%. When PFA was

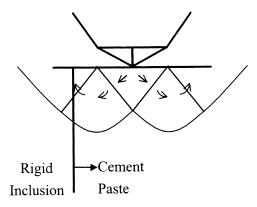


Fig. 7. Schematic description of the process that may reduce the penetration of an indenter near a rigid inclusion surface (after Igarashi [16]).

incorporated, the increments in ITZ range were reduced. For concrete made with 25% PFA by weight of total binders, the weak zone was extended by 30%. The presence of PFA resulted in a beneficial effect when PFA was added to replace 55% of OPC by weight — no obvious increase in width of the weak zone was observed for high volume PFA concrete made with a w/c ratio of 0.5; and the increase for C-0.3-55 concrete was no more than 20%. Increased micro-

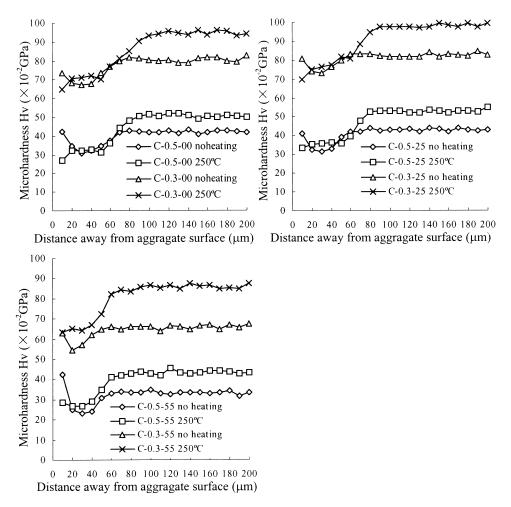


Fig. 6. Microhardness profiles in the ITZ in PFA concrete subjected to elevated temperatures.

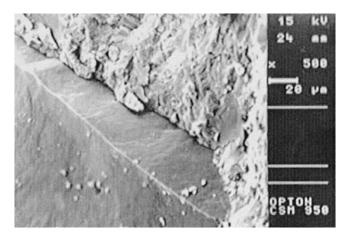


Fig. 8. SEM on the interface between aggregate and paste in C-0.3-25 concrete sample exposed to 250°C.

hardness values in the vicinity of the aggregate were observed in all unheated specimens, and these may be due to the presence of stiff inclusions in the excited range around the indentation, which restrains the flow of material under the indentation. A schematic description of the process is given in Fig. 7 to interpret the smaller penetration of an indenter near a rigid inclusion [16]. After the concretes were exposed to 250°C, further decreased Hv values in the vicinity of the aggregates were observed in all the concretes except C-0.5-55. This typical deterioration in ITZ may indicate the separation of hcp from the aggregate, as evidenced from scan electronic microscope (SEM) observations (Fig. 8). The exceptional results for C-0.5-55 concrete may be ascribed to lower hcp hardness causing a larger influencing range. In addition, improvement of microhardness within the ITZ was only obvious in concrete made with 55% PFA only, which confirms the superior fire resistance of high volume PFA concrete.

Microhardness test results on bulk pastes exposed to high temperatures are shown in Fig. 9. The trends of microhardness versus exposure temperature curves were quite similar to those of the compressive strength-exposure temperature curves shown in Fig. 3. However, the percentage rises in Hv values at 250°C were even higher than the percentage

improvements in compressive strengths. When the exposure temperatures were raised to 450°C, the Hv values were still almost equal to the results on unheated specimens, while the Hv values in high content PFA concrete were higher than the original values by 6–8%. Microhardness dropped only when the exposure temperature was as high as 650°C, at which temperature decomposition of C–S–H and CH takes place and the hydrates lose their binding properties [21–23].

The pore structure of concrete has been recognized as an important characteristic influencing the properties of the concrete such as the strength, durability and permeability [24,25]. MIP incorporated in this research is a well-developed technique that can provide information about pore structure, including the porosity and pore size distribution. As shown in Fig. 10, the total porosity of hcp increased by only about 1-2% after exposure to 250°C with an insignificant change of average pore diameter for all concretes studied. The coarsening of hcp mainly occurred after concretes had been exposed to the higher temperatures of 450°C and 650°C. The porosity of hcp increased by about 4% after exposure to 450°C and by around 10% for 650°C. It can also be seen that the average pore diameter dramatically enlarges to two to three times the original size after concrete has been exposed to 450°C and 650°C. In comparison, concrete made with a dosage of 55% PFA replacement resulted in a smaller increase in the total porosity of hcp after exposure to 450°C and 650°C. This is in keeping with the residual compressive strength test results. However, the severe permeability deterioration caused by exposure to 250°C cannot be simply due to the rise in total porosity, since the coarsening of hcp was not significant.

## 4.3. Mechanism of deterioration of PFA concrete subjected to high temperatures

When concretes are exposed to high temperatures, there are changes in the mechanical properties and the durability of concrete. However, the mechanisms causing the changes in properties is quite complex as a result of the concurrence of chemical and physical changes in hcp, aggregate, and at the interfaces.

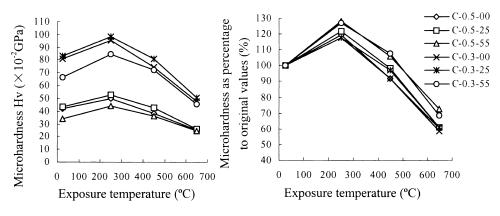


Fig. 9. Microhardness in hcp and percentages of original values.

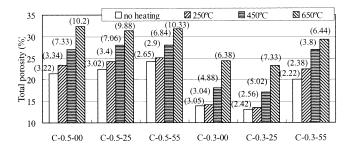


Fig. 10. Total porosity and average pore diameter of hcp in concrete (average pore diameters are marked on top of the columns in  $10^{-2}$  µm).

After concretes were exposed to a temperature of 250°C, an increase in compressive strength was observed for all mixes, with an even more pronounced increase of microhardness in the bulk hcp, but the durability of concretes was degraded. The increase of compressive strength can be partially due to the strengthened hcp during the evaporation of free water, which leads to greater Van der Waal's forces as a result of the cement gel layers moving closer to each other [10,26]. Because transportation of moisture in concrete is rather gradual, residual moisture in concrete allowed accelerated hydration at the early stage of heating concretes to high temperatures. Further hydration of cementitious materials is another important cause of the hardening of hcp. Especially for PFA concrete, unhydrated PFA particles can react with calcium hydroxide and produce C-S-H like gels [21,26]. The hardening effect is compatible with the microhardness tests results on the bulk pastes. However, weakening of the ITZ between aggregate and hcp also occurred during the exposure to 250°C. At the same time, the microhardness profiles in the ITZ showed further decrease of Hv around aggregates, indicating that tiny cracks may have initiated along the boundary, that was supported by the SEM observations shown in Fig. 8. At such an exposure temperature, coarsening of the pore structure of cement paste also occurred. Fortunately, the hardening effect can compensate for the loss of strength caused by the coarsening of the cement paste and the weakening of the ITZ. With respect to the durability of concrete exposed to 250°C, the weakened transition zone played the predominant role in the nearly doubled values observed in the rapid chloride diffusion test. Since microcracks might have developed along the boundary due to the swelling of physically bound water layers and thermal incompatibility between aggregates and cement pastes [27,28], the path for ion penetration was shortened [29,30]. In addition, increased porosity in the hcp also made penetration of chloride easier.

During exposure to 450°C, coarsening of hcp occurred with a concurrent drop in hardness. Dehydration became the predominant influence although thermal hydration played a more effective role in case of heating to 250°C [26]. Microcracking also increased significantly beyond 300°C [31,32], which is responsible for the further degradation of durability for specimens heated to 450°C and 650°C. The

gain of strength after heating to 250°C was lost after concretes were heated to 450°C. The loss of compressive strength was in good agreement with the decrease in microhardness in bulk pastes when exposure temperatures were raised from 250°C to 450°C. Decomposition of the major hydrate, known as C-S-H, was inevitable as the exposure temperature was raised to 650°C [22,23], causing severe coarsening in its microstructure of pastes and the loss of binder property. Hence sharp drops in microhardness values and compressive strengths were observed in this research together with a significant increase of chloride diffusion in concretes. Disintegration of aggregates might also occur when concretes are heated to 800°C [4]. At the same time, dehydration went further at such an exposure temperature. It is therefore commonly agreed that concretes can only maintain a minor part of their original compressive strength after exposure to 800°C [6]. The chloride diffusion tests could not proceed because the cracking is too severe and assessing the durability of concretes exposed to such a high temperature becomes meaningless.

Microcracking was found to be a major cause of deterioration when concretes were exposed to high temperatures, which was reported to initiate around Ca(OH)<sub>2</sub> crystals and then around unhydrated cement particles [33]. From Table 3 (after Lam [34]), it is reasonable to assume that PFA concretes are less susceptible to cracking during heating because PFA can consume Ca(OH)<sub>2</sub> by further hydration [35,36]. Ca(OH)<sub>2</sub> would decompose after exposure to above 400-600°C [21], and the rehydration of dissociated Ca(OH)<sub>2</sub> becomes a detrimental cause of cracking, which is accompanied by a 44% volume increase [9]. It was also reported that bound water in OPC-fly ash paste was more than twice that in OPC paste [37], and the released water can also help the fire resistance of concrete. It can be seen from the test result that the addition of PFA into binders could provide concretes with better residual properties after exposure to temperatures from 250°C to 650°C. The residual compressive strengths of high PFA content concretes remained remarkably close to their original values. Thus it may be feasible to increase the allowable "working" temperature for conventional concrete by incorporating a high volume of PFA into concrete mixes, which means that a larger proportion of structures exposed to elevated temperatures can remain serviceable and repairable.

Ca(OH)<sub>2</sub> content of PC and fly ash cement paste (based on ignited weight, after Lam [34])

	Fly ash	Ca(OH) <sub>2</sub> content (%)				
w/b	replacement (%)	7 days	28 days	90 days		
0.3	0	11.8	12.55	12.83		
	25	9.08	8.84	6.98		
	55	5.50	4.05	3.03		
0.5	0	15.32	14.79	15.78		
	55	6.83	4.79	3.30		

### 5. Conclusions

A peak residual compressive strength higher than the original unheated value was observed for all concrete specimens exposed to 250°C, an effect which was enhanced by the inclusion of PFA. Even when the maximum exposure temperature was raised to 450°C, the strength reductions were as little as 4–15%. After exposure to a higher temperature of 650°C, however, only about half of the original strengths could be obtained. The compressive strength of concrete deteriorated even more when the exposure temperature was raised to 800°C, and the residual strengths were only about 20% to 30% of the values obtained from unheated specimens.

Deterioration of durability turned out to be the main concern, however, when concrete was subjected to the medium high temperature of 250°C. Durability deteriorated at this temperature, even though no loss of strength was observed. When concrete was subjected to higher temperatures, both strength and durability of concrete deteriorated.

Microhardness testing results revealed that the width of the ITZ increased after concretes were exposed to 250°C. The increment was roughly by 40% or 30%, respectively, for concrete made with OPC only and for concrete made with 25% PFA. Nevertheless, no obvious increase in the width of the ITZ was observed in high volume PFA concrete made with a w/c ratio of 0.5, and the increases for C-0.3-55 concrete were smaller than 20%. Tiny cracks along the transition zone might have appeared after the exposure to 250°C, resulting in changes to the microhardness profiles.

The trend curves of microhardness versus exposure temperature were similar to those of compressive strength–exposure temperature curves. However, residual microhardness values were relatively higher than those for strengths by about 10% when expressed as a percentage of the original values on tested unheated specimens. Significant losses in microhardness could only be observed in specimens exposed to 650°C. Among the concretes investigated in this research, high PFA content concretes showed the best microhardness result.

The total porosity of hcp increased little after concretes were subjected to a maximum temperature of 250°C although enlargement of the average pore diameter occurred at the same time. The coarsening of hcp became obvious when the exposure temperature was 450°C and became even more severe after exposure to a temperature of 650°C although, again, a beneficial effect of high PFA dosage was also demonstrated for the two temperatures of 450°C and 650°C.

The beneficial effect of PFA on the residual strength of concrete was still noticeable when exposure temperatures were 450°C or 650°C, which was further confirmed by the microhardness test results. Concrete made with a dosage of 55% PFA exhibited a residual strength higher than other concretes by about 10% after being subjected to the two

peak temperatures. In general, the addition of PFA contributed more or less to the residual strength of concrete for all the concrete mixes. Thus, as a broad practically oriented conclusion, the "working" temperature can be raised to 450°C if PFA is incorporated and if we are concerned primarily with a compressive strength criterion. The beneficial influence of PFA can be ascribed to the pozzolanic reaction consuming Ca(OH)<sub>2</sub> in the hydrates.

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### References

- [1] F.C. Lea, The effect of temperature on some of the properties of materials, Engineering 110 (2852) (1920) 293–298.
- [2] K.W. Nasser, H.M. Marzouk, Properties of mass concrete containing fly ash at high temperatures, Am. Concr. Inst., J. 76 (4) (1979) 537–550.
- [3] S. Ghosh, K.W. Nasser, Effects of high temperature and pressure on strength and elasticity of lignite fly ash and silica fume concrete, ACI Mater. J. 93 (1) (1996) 51–60.
- [4] Z.P. Bazant, M.F. Kaplan, Concrete at High Temperatures: Material Properties and Mathematical Models, Longman Group, UK, 1996.
- [5] B.N. Grainger, Concrete at High Temperatures, Central Electricity Research Laboratories, UK, 1980.
- [6] R. Sarshar, G.A. Khoury, Material and environmental factors influencing the compressive strength of unsealed cement paste and concrete at high temperatures, Mag. Concr. Res. 45 (162) (1993) 51–61.
- [7] F.C. Lea, R.A. Stradling, Resistance to fire of concrete and reinforced concrete, Engineering 114 (2959) (1922) 341–344 Engineering 114 (2960) (1922) 380–382.
- [8] T. Harada, J. Takeda, S. Yamane, F. Furamura, Strength, elasticity and thermal properties of concrete subjected to elevated temperatures, in: International Seminar on Concrete for Nuclear Reactors, Am. Concr. Inst., SP 1 (34) (1972) 377–406 (Detroit).
- [9] A. Petzold, M. Rohr, Concrete for High Temperatures, Maclaren and Sons, London, 1970.
- [10] W.P.S. Dias, G.A. Khoury, P.J.E. Sullivan, Mechanical properties of hardened cement paste exposed to temperatures up to 700C (1292F), ACI Mater. J. 87 (2) (1990) 160–166.
- [11] Y.N. Chan, G.F. Peng, M. Anson, Residual strength and pore structure of high-strength concrete and normal strength concrete after exposure to high temperatures, Cem. Concr. Compos. 21 (1) (1999) 23–27.
- [12] X. Luo, W. Sun, S.Y.N. Chan, Effect of heating and cooling regimes on residual strength and microstructure of normal strength and highperformance concrete, Cem. Concr. Res. 30 (3) (2000) 379–383.
- [13] ASTM C1202, Standard test method for electrical indication of concrete's ability to resist chloride ion penetration, C1202-97, in: Annual Book of ASTM, Vol. 04.02, 1999.
- [14] BS 1881-116:1983, Testing concrete: Method for determination of compressive strength of concrete cubes.
- [15] ASTM E384-99 Standard test method for microindentation hardness of materials, ASTM E384-99, in: Annual Book of ASTM, Vol. 03.01 1999.
- [16] S. Igarashi, A. Bentur, S. Mindess, Microhardness testing of cementitious materials, Adv. Cem. Based Mater. 4 (2) (1996) 48–57.

- [17] R.K. Dhir, E.A. Byars, Pulverized fuel-ash concrete: Intrinsic permeability, ACI Mater. J. 90 (6) (1993) 571–580.
- [18] T.R. Naik, S. Singh, B. Ramme, Mechanical properties and durability of concrete made with blended fly ash, ACI Mater. J. 95 (4) (1998) 454–462.
- [19] J.J. Beaudoin, R.F. Feldman, A study of mechanical properties of autoclaved calcium silicate systems, Cem. Concr. Res. 5 (2) (1975) 103-118
- [20] S. Igarashi, M. Kawamura, Effects of a size in bundled fibers on the interfacial zone between the fibers and the cement paste matrix, Cem. Concr. Res. 24 (4) (1994) 695–703.
- [21] H.F.W. Taylor, The Chemistry of Cements, Academic Press, London, 1964.
- [22] G.A. Khoury, B.N. Grainger, P.J.E. Sullivan, Transient thermal strain of concrete: Literature review, conditions within specimen and behavior of individual constituents, Mag. Concr. Res. 37 (132) (1985) 131–144.
- [23] G.F. Peng, Evaluation of fire damage to high performance concrete, PhD thesis, the Hong Kong Polytechnic University, 1999.
- [24] D.M. Roy, G.R. Gouda, Porosity-strength relation in cementitious materials with very high strengths, J. Am. Ceram. Soc. 56 (10) (1973) 549-550.
- [25] E.J. Garboczi, Permeability, diffusivity, and microstructural parameters: A critical review, Cem. Concr. Res. 20 (4) (1990) 591–601.
- [26] G.A. Khoury, Compressive strength of concrete at high temperatures: A reassessment, Mag. Concr. Res. 44 (161) (1992) 291–309.
- [27] H.L. Malhotra, Effect of temperature on the compressive strength of concrete, Mag. Concr. Res. 8 (23) (1956) 85–94.

- [28] R. Blundell, C. Dimond, R. Browne, Properties of concrete subjected to elevated temperatures, in: Technical Note No. 9, June, CIRIA Underwater Engineering Group, London, 1976.
- [29] J.P. Ollivier, M. Massat, The effect of the transition zone on transfer properties of concrete, in: J.C. Maso (Ed.), Interfacial Transition Zone in Concrete, RILEM Report II (1996) 117–131.
- [30] K.J. Wang, T. Igusa, S.P. Shah, Permeability of concrete relationship to its mix proportion, microstructure and microcracks, in: Materials Science of Concrete: The Sidney Diamond Symposium, American Ceramic Society, Westerville, OH, 1998, pp. 45-54.
- [31] W.M. Lin, T.D. Lin, L.J. Powers-Couche, Microstructures of fire-damaged concrete, ACI Mater. J. 93 (3) (1996) 199–205.
- [32] M.A. Riley, Possible new method for the assessment of fire-damaged concrete, Mag. Concr. Res. 43 (155) (1991) 87–92.
- [33] J. Piasta, Heat deformations of cement phases and the microstructure of cement paste, Mater. Struct. 17 (102) (1984) 415–420.
- [34] L. Lam, A study of high volume fly ash concrete, PhD thesis, the Hong Kong Polytechnic University, 1999.
- [35] E.E. Berry, R.T. Hemmings, M.H. Zhang, B.J. Cornelious, D.M. Golden, Hydration in high-volume fly ash concrete binders, ACI Mater. J. 91 (4) (1994) 382–389.
- [36] R.F. Feldman, G.G. Carette, V.M. Malhotra, Studies on mechanism of development of physical and mechanical properties of high-volume fly ash concrete, Cem. Concr. Compos. 12 (1990) 245–251.
- [37] E.E. Berry, R.T. Hemmings, B.J. Cornelius, Mechanisms of hydration reactions in high volume fly ash pastes and mortars, Cem. Concr. Compos. 12 (1990) 253–261.