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EFFECT OF SILICA FUME ON THE PHASE COMPOSITION AND MICROSTRUCTURE OF THERMALLY TREATED CONCRETE

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ABSTRACT

The influence of elevated temperature on the microstructure of concrete is vital for fire-resistance studies. Silica fume tends to improve ordinary concrete properties, due to its reaction with hydrated products.This study aimed to investigate experimentally the change occurring in the phase composition and microstructure of concrete pastes containing silica fume after heat treatment. The temperature varied from 100 to 600°C by increment of 100°C for three hours period. The investigation was performed by means of the X-ray diffraction analysis, differential thermal analysis and scanning electron microscope. The results of this investigation show that, additional hydration of unhydrated cement grains, recrystallization deformation and transformation of CSH phases were occurred.

Introduction

Cases of natural deterioration of building materials such as concrete when exposed to fire is frequently reported from different parts of the world. Deterioration can often reach a level at which the structure may have to be thoroughly renovated or completely replaced. An effective estimation of concrete resistance to high temperatures required a profound knowledqe of phenomena taking place during exposure to heat. These phenomena are connected directly with physico-chemical processes occurring in the components and are brought about by a temperature increase. The most important processes are chemical reactions, phase transformations and heat deformations occurring in the microscopic scale in particular phases of concrete components [1]. Silica fume has been shown to improve the microstructure of cement paste in concrete by densifying the cement paste matrix and the porous paste-aggregate interfacial zone [2-4]. The porosity of silica fume concrete decreases as the silica fume content increases up to 5% and then increases as the silica fume content increases [5]. The replacement of ordinary Portland cement by 10% silica fume improve the physical and mechanical properties of concrete when exposed to elevated temperature [6]. Therefor the effect of silica fume on the microstructure and phase composition of concrete is important for fire-resistance studies.

Experimental Work

The concrete specimens were made with a cement : sand : aggregate ratio of 1 : 2 : 3 by weight and ab water / (cement + silica fume) ratio of 0.40. The ordinary Portland cement has been partially replaced by silica fume(SF) of ratios 10, 20 and 30% by weight. After 28 days of moist curing, concrete specimens were dried at a temperature of 105 ± 5°C for 24 hours in electric furnace. Next they kept for 3 hours in each of the investigated temperature, in the range of 200°C to 600°C in intervals of 100°C. The concrete samples were cooled together with the furnace to the indoor room air temperature and they were applied to structural tests. The crystalline phases present in the hydrated product were identified using X-ray diffraction(XRD) techniques. Nickle-filtered Cu-Kα radiation at 40 KV and 20 mA was used. The differential thermal analysis(DTA) was used for observation of the thermal deformation course in the range of investigated temperature using DT-30 thermal analyzer. The scanning electron microscope (JSM-T20) was used for identification of the changes occurring in the microstructure of the formed and/or decomposed phases.

Results and Discussion

Silica fume is known to improve both of the mechanical characteristics and durbility of concrete. The principal physical effect of silica fume in concrete is as a filler, which, can fit into spaces between cement grains [7]. The chemical reactions of silica fume is due to its high surface area and high content of amorphous silica. The high-strength concrete which produced with condensed silica fume appears to be more prone to spalling in a fire than normal strength concrete [8]. The results of the X-ray diffraction and DTA investigations into the phase content of Portland cement pastes, with and without silica fume, exposed to heat are shown in Figures (1-4).

Figure(1) shows the X-ray diffraction patterns of the hardend concrete samples, prepared with water/cement(W/C) ratio of 0.40 and hydrated for 28 days. The concrete samples were treated at various temperatures of 105, 200, 400 and 600°C. All thermally treated samples contain quartz, calcium silicate hydrate(CSH), calcium hydroxide[$Ca(OH)_2$] and calcium carbonate ($CaCO_3$). The intensity of the peaks caracteristic for CSH (d=3.23°A) and $Ca(OH)_2$ (d=4.9°A) phases increased in the sample treated at 200°C and then decreases in the sample treated at 400 and 600°C. The increase in intensity of $Ca(OH)_2$ peaks at 200°C is a result of recrystallization of a morphous $Ca(OH)_2$ in addition of the formation of larger amounts of $Ca(OH)_2$, but the increase in intensity of CSH peaks is mainly due to additional hydration of unhydrated cement. At 400°C, however, there appeared a complete disappearance of the CSH peaks indicating the complete decomposition of calcium silicate hydrates; in addition, the intensity of the peaks characterizing the $Ca(OH)_2$ phase decreased, indicating a partial decomposition of $Ca(OH)_2$. At 600°C, all the XRD peaks characteristic for the hydration products are completely disappeared.

Figure(2) demonstrates the X-ray diffraction of heat treated concrete samples, made with a W/(C+SF) ratio of 0.40 and containing 10% silica fume. All peaks characterising the portlandite phase[$Ca(OH)_2$] are completely disappears. The calcium hydroxide released during cement hydration is acutully consumed as a result of interaction with active silica fume to form the CSH phases. The CSH-peaks are also completely disappeared at higher temperature of thermal treatment. In the temperature range 200 - 600°C the carbonization of $Ca(OH)_2$ increases. This is indicated by a decrease in the $Ca(OH)_2$ content in favour of the $CaCO_3$

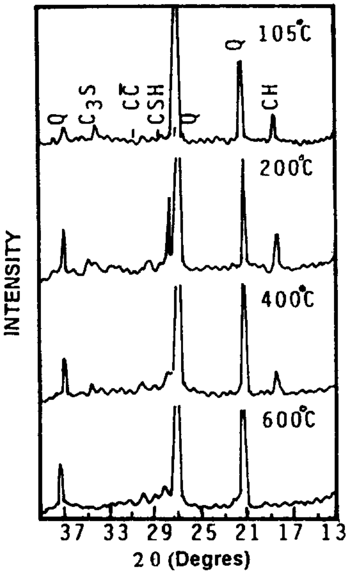


FIG. 1.
X-ray diffraction of heat treated concrete at various temperature.

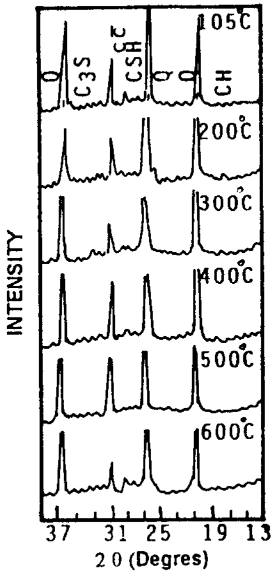


FIG. 2.
X-ray diffraction of heat treated concrete containing 10% SF.

CH	calcium hydroxide
CSH	calcium silicate hydrate
CC	calcium carbonate
C_3S	tricalcium silicate
Q	quartz

increase observed by of X-ray diffraction method (Figs. 1 and 2). This portion of $Ca(OH)_2$ which did not undergo carbonization, is decomposed in the temperature range 450 - 550°C with emission of calcium oxide [1].

Figure(3) shows the DTA thermograms of the various heat treated concrete samples. The results indicate the existence of four endothermic peaks. The first broad peak located at the temperature range of 140-230°C is mainly due to the decomposition of CSH. The second endothermic peak observed at about 480°C represents the decomposition of calcium hydroxide. The third endothermic located at about 575°C describing the quartz conversion. The fourth endotherm observed at about 720°C represents the thermal decomposition of small amounts of $CaCO_3$. The peak area of the CSH phases increases as the temperature of the thermal treatment increases up to 200°C; then, it decreases, or even diminishes as the temperature increases up to 600°C. The initial increase in the peak area with increasing temperature up to 200°C is mainly attributed to the further hydration reaction between the unhydrated partes of the cement grains and the residual free water located in the remaining pore system of the concrete specimen. The sharp decrease in the peak area of CSH phases at 400 and 600°C is due to the complete decomposition of CSH in this temperature range. The peak area of $Ca(OH)_2$ phase is nearly stable for the concrete sample treated at 105, 200 and 400°C; then the $Ca(OH)_2$-peak shows a sharp decreas at 600°C. The decrease of $Ca(OH)_2$ in the concrete sample treated at 600°C is owing to the decomposition of $Ca(OH)_2$ phase in the temperature range 450-550°C.

Figure(4) illustrates the DTA thermograms of heat treated concrete samples made with an initial W/(C+SF) ratio of 0.40 by weight and containing 10% silica fume.The results indicate the existence of the four distinct endothermic peaks obtained in case of the concrete samples

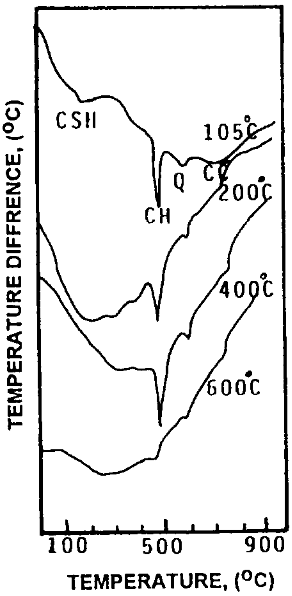


FIG. 3.
DTA thermograph of various heat treated concrete after hydration for 28 days.

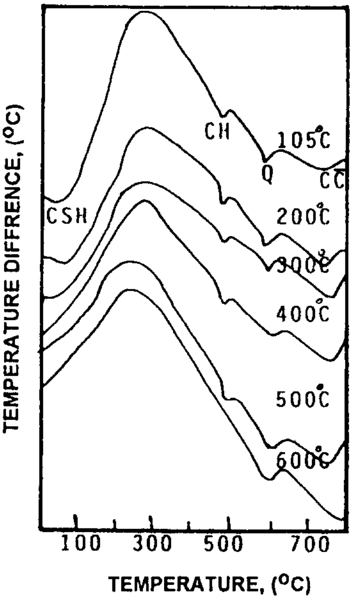
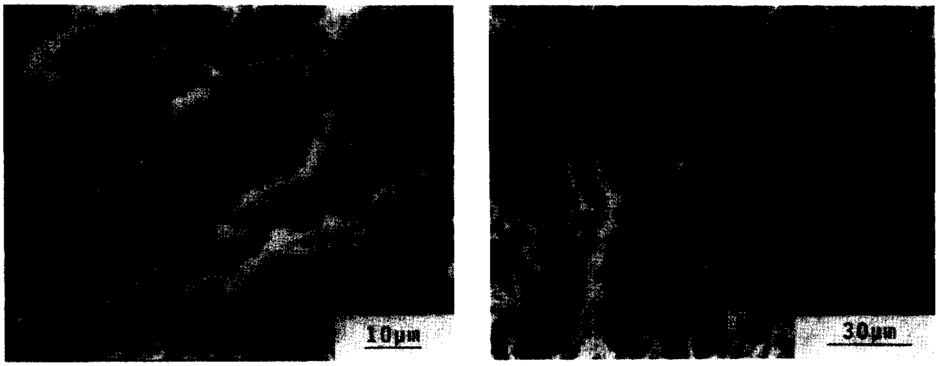


FIG. 4.
DTA thermograph of various heat treated concrete containing 10% silica fume.

made without silica fume with two main basic differences, namely; (i) the disappearance of the endotherms located at about 480°C characterising the calcium hydroxide phase and (ii) the marked intensification of the endotherm located at the temperature range 110-140°C.

The micro-structure of pastes observed by scanning electron microscopy indicated that, the characteristic microcracks in the paste structure, caused by different heat deformations of phases, are shown in figures 5, 6 and 7. The well-crystallized calcium hydroxide appeared as stacked parallel layers as will as ill-crystallized calcium silicate hydrate as shown in Fig.(5). These hydrates appeared as clear binders between the concrete constituents. It also illustrates



Thermally treated at 105°C; x1000

Thermally treated at 600°C; x500

FIG. 5.
SEM micrograph of concrete (W/C=0.04).

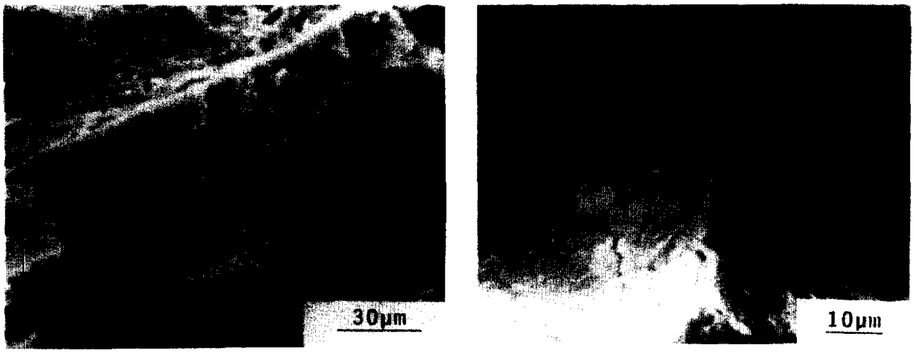
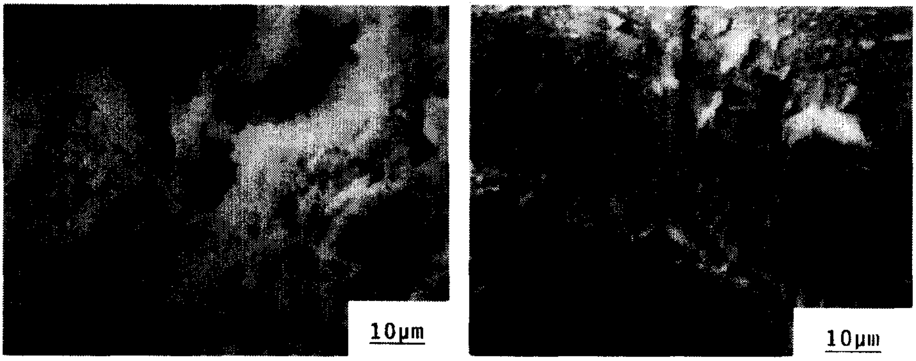


FIG. 6.
SEM micrograph of concrete (W/(C + SF) = 0.04) containing 10%SF.

the microstructure obtained after thermal treatment at 600°C, a complete disruption of the previously built structure with appearance of several microcracks were observed.

The essential feature of all SEM micrographs in Fig.(6) and Fig.(7) for concrete containing silica fume is a more dense structure. The cement hydrates as well as the CSH phases produced as a result of consumption of free $Ca(OH)_2$ by active silica fume are deposited within the pore system and around the grains of the concrete constituents leading to a more dense structure of concrete specimens containing silica fume. Concrete containing 10% silica fume is perfectly stable upon thermal treatment up to 300°C; but at higher temperatures (up to 600°C) microcracks are first appeared and enlarged. With increasing silica fume content of concrete to 20% and 30%, however, the microcracks are appeared after thermal treatment at 105°C and later enlarged with increasing temperature up to 600°C. Therefore, the structure stability is reasonably high by addition of 10% silica fume and the thermal stability decreases at higher silica fume contents (20 and 30%).



Thermally treated at 105°C; x1000 **Thermally treated at 600°C; x1000**

FIG. 7.
SEM micrograph of concrete (W/(C+SF)=0.04) containing 30%SF.

Conclusions

The main conclusions derived from this study may be summarized as follows :

1- The calcium hydroxide phase is nearly stable for the concrete (W/C=0.40) sample treated at 105, 200 and 400°C; then its stability decreases as the temperature increases up to 600°C due to the decomposition of $Ca(OH)_2$ phase in the temperature range 450-550°C.

2- The thermal treatment of concrete (W/C=0.40) from 400 to 600°C leads to an almost complete decomposition of the main hydrates with appearance of several microcracks.

3- The calcium carbonate phase ($CaCO_3$) begins to decompose from 600°C upwards.

4- The addition of silica fume to ordinary concrete leads to the consumption of $Ca(OH)_2$ obtained during cement hydration as a result of interaction with active silica fume to form the CSH phases.

5- Concrete specimens containing 10% silica fume are stable for thermal treatment up to 300°C.

6- The increase of silica fume ratios (20 and 30%) leads to appearance of microcracks after thermal treatment at 105°C and these were later enlarged with increasing temperature up to 600°C.

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