ACI MATERIALS JOURNAL TECHNICAL PAPER

Title no. 99-M39

Effects of Metakaolin and Silica Fume on Properties of Concrete

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Metakaolin is a relatively new mineral admixture for concrete. It is comparable to silica fume in pozzolanic reactivity, but is lower in price. The effects of metakaolin and silica fume on various properties of concrete were investigated and compared in this study. Seven concretes were cast at a water/binder ratio of 0.35 with 0, 5, 10, and 15% cement replaced by either metakaolin or silica fume. The concretes were tested for slump, compressive strength, free shrinkage, restrained shrinkage cracking, and chloride diffusivity by ponding. Metakaolin-modified concrete showed a better workability than silica fume-modified concrete. As the replacement level was increased, the strength of the metakaolin-modified concrete increased at all ages similarly to that of the silica fume-modified concrete. Both mineral admixtures reduced free drying shrinkage and restrained the shrinkage cracking width. However, the cracking time was earlier for these two concretes. The two admixtures also greatly reduced the chloride diffusivity of the concrete.

Keywords: cracking; shrinkage; silica fume; slump; strength.

INTRODUCTION

Commercially available since the mid-1990s, high-reactivity metakaolin (MK) is one of the recently developed supplementary cementing materials for high-performance concrete. It is produced by calcining purified kaolinite clay at a specific temperature range to drive off the chemically bound water in the interstices of kaolin and destroy the crystalline structure, which effectively converts the material to the MK phase, which is an amorphous aluminosilicate. Unlike industrial by-products, such as silica fume (SF), fly ash, and blast-furnace slag, MK is refined carefully to lighten its color, remove inert impurities, and control particle size. This well-controlled process results in a highly reactive white powder that is consistent in appearance and performance. The particle size of MK is generally less than 2 μm, which is significantly smaller than that of cement particles, though not as fine as SF. It is typically incorporated into concrete to replace 5 to 20 wt% of cement. MK improves concrete performance by reacting with calcium hydroxide to form secondary C-S-H. Because of its white color, high-reactivity MK does not darken concrete as SF typically does (the white-colored SF is very limited in tonnage), which makes it suitable for color-matching and other architectural applications. Research has shown that mixtures containing high-reactivity MK yield comparable performance to SF mixtures in terms of strength, permeability, chemical resistance, and drying shrinkage resistance.[1-7]

MK was found to improve concrete properties while offering good workability. Concrete with MK requires 25 to 35% less high-range water-reducing admixture than concrete with silica fume to achieve a comparable slump of 12 to 18 cm at a water/binder ratio of 0.36 to 0.38.[1-2] MK-modified concrete has a creamier texture, sets somewhat faster, generates less bleed water, and has a better finishability than concrete with SF.[1,8] However, there are also some different conclusions.[3,4]

The compressive and flexural strength developments of the MK mixtures are significantly faster than those of the nonpozzolanic control mixture, and slightly higher than or equivalent to those of the SF mixtures.[1-2] For concretes with the same water/binder ratio, compressive strengths increased dramatically at all ages with increased replacement percentage by mass of high-reactivity MK.[6,9] MK is particularly effective in improving the post-peak energy absorption capacity for concrete with fibers. Unlike SF, no particular post-peak brittleness occurs.[10] MK is also more effective than SF in improving the pullout performance of deformed steel fibers embedded in cement-based matrixes.[11]

The long-term drying shrinkage of MK mixtures is less than that of plain concrete and similar to that of the SF mixtures.[1] According to the results on pastes reported by Wild, Kahtib, and Roose,[7] chemical shrinkage was found to increase for compositions with up to 15% MK replacement and then decrease for compositions with a replacement greater than 15%. Expansion by curing underwater showed little variation for compositions with up to 10% MK replacement, and then increased for compositions with a replacement greater than 15%. Kinuthia et al.[12] reported a reduced autogenous shrinkage at later ages (up to 200 days) of the cement paste with 20% MK replacement. But according to Brooks and Johari,[13] at a 5% replacement level, the addition of MK increased the total autogenous shrinkage considered from the time of initial set, while at replacement levels of 10 and 15%, it reduced the total autogenous shrinkage.

Kostuch, Walters, and Jones[14] demonstrated that MK is particularly effective in reducing the rate of diffusion of sodium and chloride ions. The diffusion coefficient was reduced by approximately 50% when 8% MK was added to the concrete mixture.[6,9] The chloride permeability of the MK mixture, measured according to ASTM C 1202, was much lower than that of plain concrete,[3] and was slightly higher[2] or lower [1] than that of the SF concrete. Moreover, both MK and SF concretes had a very low permeability rating according to ASTM C 1202. According to both bulk-diffusion tests and modified AASHTO T259 tests, replacing 8% cement with the high-reactivity MK in a 0.40 water/binder ratio concrete improved diffusion characteristics as much or more than a reduction of the water/binder ratio to 0.30.[9] Dhir and Jones[15] have shown that using ternary blends of normal fly ash with SF or MK could provide

ACI Materials Journal, V. 99, No. 4, July-August 2002.
MS No. 01-261 received August 20, 2001, and reviewed under Institute publication policies. Pertinent discussion will be published in the May-June 2003 *ACI Materials Journal* if received by February 1, 2003.

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better chloride resistance for concrete than using ultrafine fly ash only.

Other aspects of durability, such as ASR and sulfate resistance, are improved as the replacement level of cement with MK increases, up to 25% replacement.[5,16] The quick consumption of CH, and the increase in the C-S-H and hydrated gehlenite (C_2ASH_8) lead to refinement of the pore structure, which was considered the principal mechanism for the improvement of concrete properties by the addition of MK.[17-19]

There is less information about the properties of MK concrete, however, than for other mineral admixture-modified concretes, and the available conclusions are somewhat contradictory. The objectives of the study were to investigate systematically the effect of MK on the properties of concrete at various replacement amounts, including workability, compressive strength, free shrinkage, restrained shrinkage cracking, and chloride diffusivity. SF was chosen for comparison because of its effective application as an ultrafine mineral admixture.

RESEARCH SIGNIFICANCE

High-reactivity MK is a supplementary cementing material developed recently for high-performance concrete. Although some works have reported that it improves concrete properties, information about the properties of metakaolin-modified concrete is still limited and somewhat contradictory, which retards its application in the construction practice. This study systematically investigated the effect of MK on concrete properties, especially free and restrained shrinkage, as well as chloride diffusivity. These results are compared with those of concrete modified with SF. It was found that MK-modified concrete had comparable performance to that of SF-modified concrete in terms of strength, shrinkage, and chloride diffusivity.

EXPERIMENTAL

Specimen preparation

Seven concrete mixtures were cast using 0, 5, 10, and 15% by mass replacement of cement with high-reactivity MK or SF, at a water/binder ratio of 0.35 and a sand-to-aggregate ratio of 40%. The mixtures were marked as PC, MK5, MK10, MK15, SF5, SF10, and SF15, respectively. For all of the mixtures, the ratio of binder (including cement, SF, and MK) to fine aggregate and to coarse aggregate was kept constant at 1:1.55:2.33. The cement used was ordinary portland cement manufactured locally, with a specific gravity of 3.15 and a fineness of 385 m^2/kg. The average powder diameter is 19.5 m. The high-reactivity MK, which was manufactured in the U.S., had a specific gravity of 2.55, a specific surface area of 12×10^4 m^2/kg, and an average particle size of 2.23 μm. The SF was a commercially available product with a specific gravity of 2.26 and an average particle size of 0.1 μm. The chemical compositions of these three powders are listed in Table 1. The coarse aggregate was a 10 mm crushed limestone with a specific gravity of 2.75 and an absorption of 1.7%. The fine aggregate had a specific gravity of 2.66, an absorption of 1.4%, and a fineness modulus of 2.3. All mixtures contained 1.0% of a naphthalene sulfonate-based high-range water-reducing admixture FDN by mass of binder and 0.25% of a retarder D17 by mass of binder. The details of the mixture proportions are presented in Table 2. Except for shrinkage tests, the specimens were cured at 25 °C and a relative humidity of over 95%.

At 3, 28, and 65 days of age, cylinder specimens, 200 mm in height and 100 mm in diameter, were tested for compressive strength with an MTS machine in accordance with ASTM C 39/C 39M.

For the shrinkage tests, each set included an unrestrained ring-type specimen to measure the free shrinkage, and a restrained specimen to record the cracking onset time and width. The restrained specimen was made of a concrete ring (35 mm in thickness and 140 mm in height) cast around the outer perimeter of a steel ring with

Table 1—Chemical composition and color of cementitious materials

Chemicals	Cement	Silica fume	Metakaolin
SiO_2	21.0%	92.9%	51.2%
Al_2O_3	5.2%	0.69%	45.3%
Fe_2O_3	2.3%	1.25%	0.60%
MgO	3.9%	1.73%	—
CaO	63.9%	0.4%	0.05%
Na_2O	0.5%*	0.43%	0.21%
K_2O		1.19%	0.16%
SO_3	2.4%	—	—
LOI	—	1.18%	0.51%
Color	Gray	Dark gray	White

*As equivalent Na_2O.

Table 2—Mixture proportions of concrete, kg/m^3

Material	C	MK	SF	W	S	G	High-range water-reducing admixture	Retarder
PC	462	—	—	162	716	1176	4.63	1.16
MK5	438	23	—	162	715	1174	4.62	1.16
MK10	415	46	—	161	714	1172	4.61	1.16
MK15	391	69	—	161	712	1170	4.61	1.15
SF5	438	—	23	162	714	1173	4.62	1.16
SF10	414	—	46	161	712	1169	4.60	1.15
SF15	390	—	68	161	710	1166	4.59	1.15

254 and 305 mm inner and outer diameters, respectively. The unrestrained ring was similar to the restrained ring, but without the steel ring.

For the shrinkage tests, the outer rings were stripped off 24 h after casting. The top surface of the concrete rings was then sealed with epoxy resin to avoid moisture loss. For the free shrinkage rings, the inner circumferential surface was also sealed. Therefore, drying was allowed only from the outer circumferential surface of the specimens. The specimens were cured for one day at 20 °C and 100% relative humidity, then stored at 23 °C and 40% relative humidity, and measured every 24 h for the first 30 days and every 3 days thereafter.

The free shrinkage was the average of five measurements by a dial-gage extensometer on five pairs of brass studs fixed on the top surface of the specimen along the circumferential direction. The onset times of new cracks on the restrained specimen were recorded. The crack width was the average of the three values measured by a microscope to the nearest 0.02 mm on the outside surface of the specimen at three positions: one-quarter, one-half, and three-quarters of the vertical distance from the top surface of the ring. If there was more than one crack on a specimen, the sum of the average values of all cracks was used as the crack width.

The resistance to chloride diffusion was determined by the diffusion-cell method. The cell consisted of two chambers, A and B. One ∅100 × 200 mm cylinder was cast for the chloride diffusion test for each concrete mixture that was cured for 3 months. Three specimens of ∅100 × 20 mm were cut from its upper, middle, and lower quarter parts, with the top and bottom casting surfaces removed. Each slice was then placed between the two chambers of the diffusion cell. Its edge was covered by O-rings and sealed with epoxy to prevent leakage. A saturated $Ca(OH)_2$ solution was poured into both chambers for five days before a 5 M NaCl solution was poured into Chamber A to avoid anomalous effects due to sorption rather than diffusion of chloride ions. The cell was kept at room temperature (approximately 23 °C), and the concentration of chloride ions in Chamber B was measured using an ion meter. After each measurement, an appropriate amount and concentration of salt solution was added into the chambers to keep the concentration of chloride ions in Chamber B from being affected by the measurement and to keep that of Chamber A constant at 5 M.

EXPERIMENTAL RESULTS AND DISCUSSIONS

Slump

The effect of MK or SF on the slump of concrete at different replacement levels is shown in Fig. 1. It can be seen that MK offered a much better workability than did SF for the given mixture proportions. Indeed, concrete mixtures with 5 to 10% MK had a slightly higher slump than the control mixture. Even when the replacement level of MK was increased to 15%, the slump was only decreased by approximately 10% and was still greater than 15 cm. For SF-modified concrete, the slump value showed only a small decrease at the replacement level of 5%. However, it decreased almost linearly with an increase of SF content to 15%. The results, also indicated by Caldarone, Gruber, and Burg[1] and Caldarone and Gruber,[2] meant that the concrete mixtures modified by MK required less high-range water-reducing admixture than SF mixtures to achieve similar workability at the same water/binder ratio. This reduction in high-range water-reducing admixture demand may result in less tendency for surface tearing during finishing operations and lead to an overall better finishability. In addition, the MK-modified mixtures may be more economical because of a lower dosage of high-range water-reducing admixture. Bai et al.[4] observed a different phenomenon, namely that the workability was substantially reduced for mixtures containing MK, with greater reductions being experienced as the MK replacement level increased. But this conclusion was drawn only from the experiments on low- to medium-slump (5 to 110 mm) concretes with a small amount of high-range water-reducing admixture or even without any.

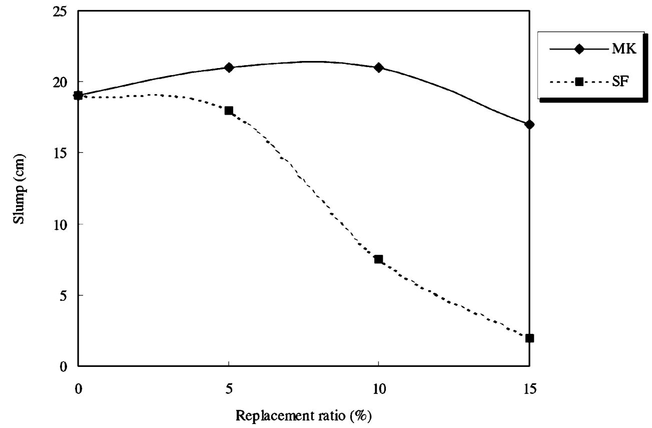

Fig. 1—Effect of metakaolin or silica fume on slump at different replacement levels by mass of cement.

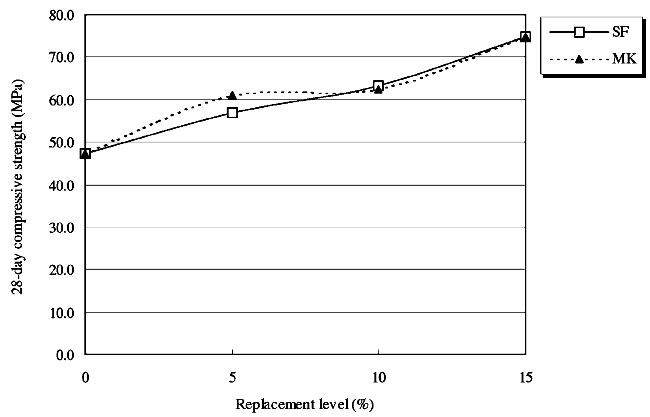

Fig. 2—Effect of metakaolin or silica fume on 28-day compressive strength of concrete at different replacement levels by mass of cement.

Compressive strength

Figure 2 demonstrates the effect of MK or SF on the 28-day compressive strength at different replacement levels. Figure 3 shows the compressive strength development of MK and SF concretes with curing age. It is clear from Figure 2 and 3 that at the same replacement level, MK increased concrete strength at all ages to almost the same extent (approximately 5 to 55%) as SF did. By increasing the replacement level from 5 to 15%, the strengthening effect of MK increased. Wild, Khatib, and Jones[20] suggested that there existed an optimum replacement level of approximately 20% to give maximum long-term strength enhancement. The compressive strengths of the concrete mixtures MK5, MK10, and MK15 were approximately 28, 38, and 45% higher than that of the control concrete at 3 days; 25, 28, and 53% higher at 28 days; and approximately 4, 16, and 21% higher at 65 days. This result was different from the

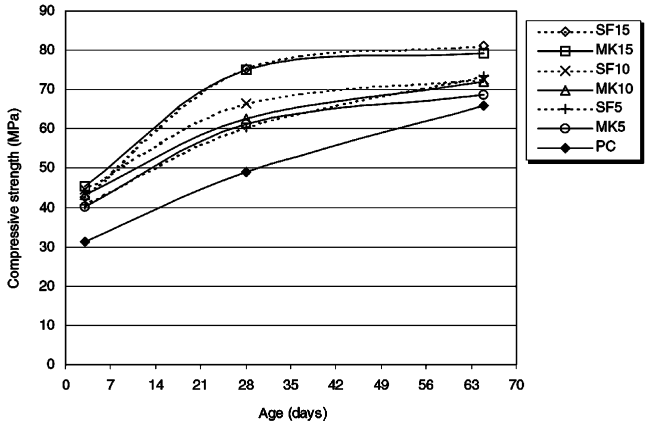

Fig. 3—Compressive strength developments of MK and SF concretes.

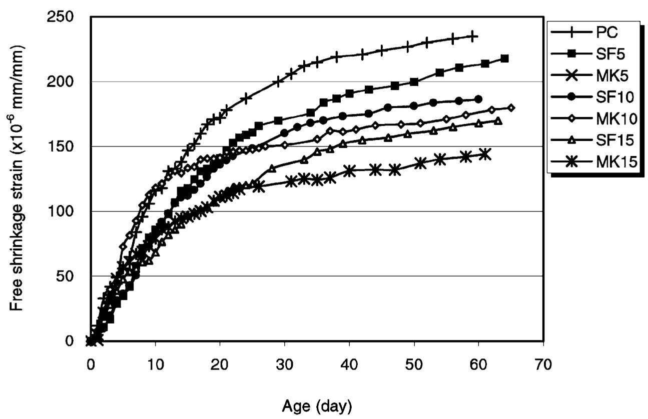

Fig. 4—Development of free shrinkage strain with time.

claim of Wild, Khatib, and Jones[20] that the contribution of MK to concrete strength was restricted beyond 14 days. But Fig. 3 did show that the potential of strength increase for 15% MK or 15% SF concrete was rather limited after the age of 28 days; the 65-day compressive strength of these two concretes only increased by approximately 6 to 8% compared with the 28-day strength.

Free shrinkage

The strain developments versus time of the free shrinkage specimens are presented in Fig. 4. The shrinkage of all of the specimens developed quickly up to approximately 3 weeks, then the rate of the shrinkage development decreased. The shrinkage of the concrete with MK or SF decreased with increasing replacement levels at 28 days and thereafter; the free shrinkages of the concrete mixtures MK5, MK10, MK15, SF5, SF10, and SF15 were approximately 15, 25, 40, 15, 22, and 33% less than that of the control concrete at 28 days, respectively. This agrees with the observations of Al-Khaja,[21] Bentur and Goldman,[22] and Alexander.[23] They concluded that the shrinkage and creep of plain concrete were considerably or moderately reduced with the incorporation of SF, showing a one-month reduction in strain of 34.9 and 18.5% for shrinkage and creep, respectively, which led to a reduction in the total deformation of 20.8%.[21] Bentur and Goldman[22] attributed this to the smaller weight loss on drying. However, there are also different conclusions in the literature.[1,7,12]

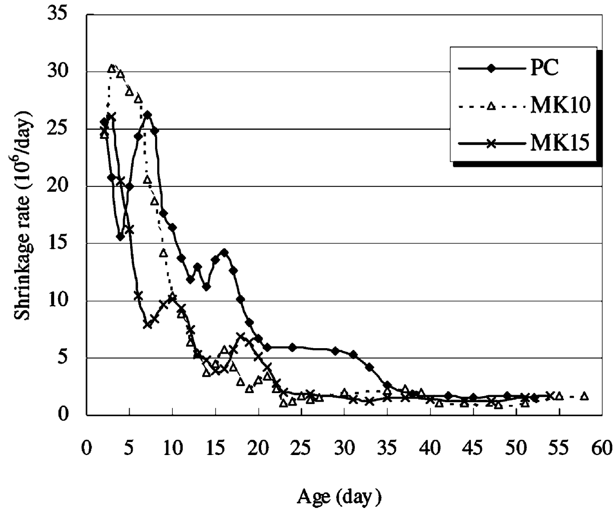

Fig. 5—Effect of metakaolin content on shrinkage rate of concrete with time.

The shrinkage rates in Fig. 4 have been calculated and shown in Fig. 5 and 6 for the MK- and SF-modified concrete, respectively. The shrinkage rates of MK10 and MK15 concrete were higher than the control concrete before the age of 4 and 5 days, respectively, and then the rates of the two MK concretes decreased and were less than those of the control concrete. The higher the MK content, the lower the highest peak of the shrinkage rate of concrete and the slower the shrinkage rate of concrete overall. Compared with the SF concrete at the same replacement level (Fig. 6), the MK concrete showed a somewhat faster development of shrinkage before the age of about one week, then a slower development after that (Fig. 5). Zhang and Malhotra [3] made a similar conclusion that the concrete with 10% MK had a lower drying shrinkage compared with that of the control and SF concretes after 7 days of initial curing in lime water.

Free shrinkage tests alone cannot offer sufficient information on the behavior of concrete structures because virtually every concrete element is restrained in some way, either by reinforcement or by the boundary condition of a structure. However, the strain obtained from a free shrinkage test can be used as the eigenstrain due to shrinkage for the corresponding restrained shrinkage test. The stress distribution due to shrinkage in the restrained specimen can be evaluated from the value of the eigenstrain and the restrained conditions of the specimen.[24]

Restrained shrinkage cracking

Two cracks each were detected in Specimens PC, SF10, and SF15 at the age of 17 and 19 days, 9 and 13 days, and 7 and 8 days, respectively. Only one crack was observed in the other four specimens. The developments of crack width with time for the restrained shrinkage specimens are shown in Fig. 7 and 8 for the MK- and SF-modified concrete, respectively. It was found that the first crack generally appeared 1 to 2 weeks after casting. Basically, the width of the first crack developed very quickly in the first few days, and then the rate of development decreased.

As mentioned previously, the ultimate free shrinkage of the SF or the MK concrete was less than that of the control portland cement concrete. Figure 7 and 8 show that the stabilized crack widths in the former two kinds of concretes were less than that in the control PC and the crack width also decreased with the increase in the SF or MK replacement level: for the

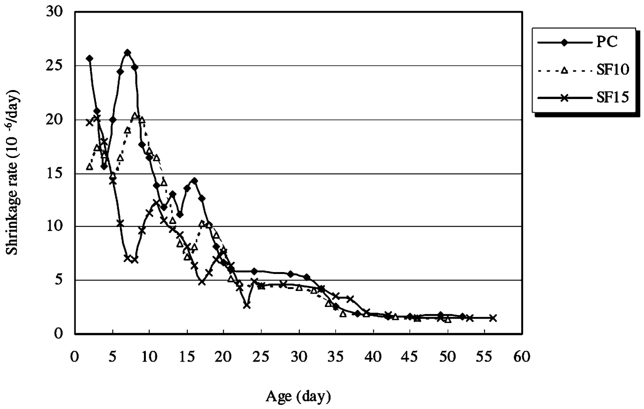

Fig. 6—Effect of silica fume content of shrinkage rate of concrete with time.

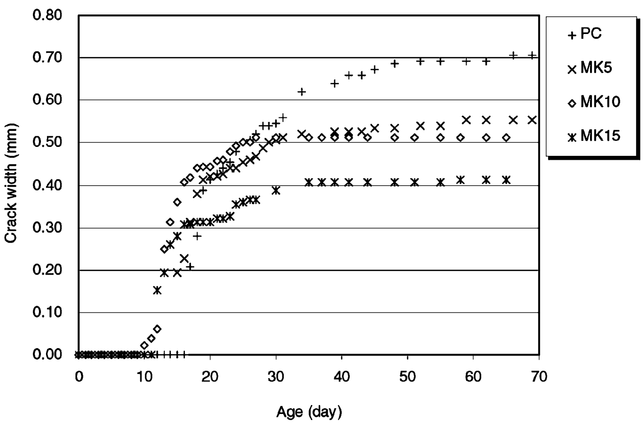

Fig. 7—Effect of metakaolin on development of crack width in restrained specimens.

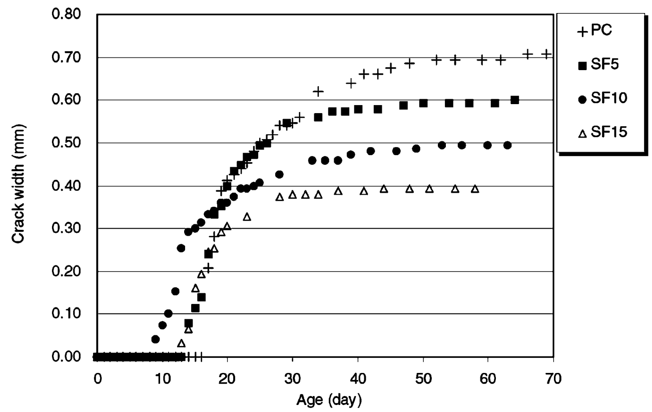

Fig. 8—Effect of silica fume on development of crack width in restrained specimens.

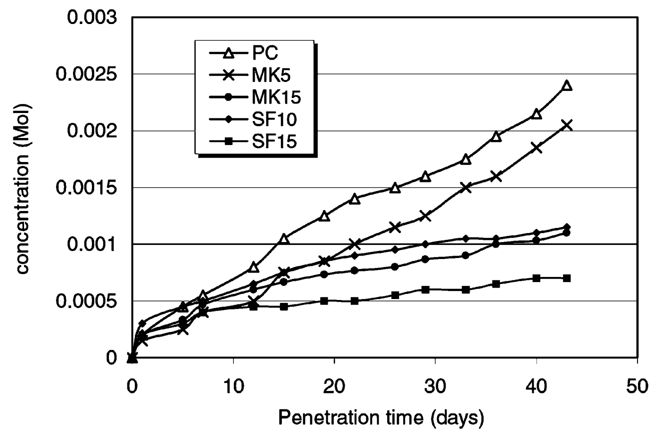

Fig. 9—Effect of metakaolin or silica fume on chloride diffusivity at different replacement levels.

PC, the stabilized crack width was 0.70 mm; for the MK5, MK10, and MK15 concretes, the result was 0.55, 0.51, and 0.41 mm, respectively; and for the SF5, SF10, and SF15 concretes, the result was 0.60, 0.49, and 0.39 mm, respectively. This difference should be partly caused by the higher bond strength between the SF or MK concrete and the steel ring. However, the onset times of cracking in the concrete with SF or MK were earlier than that in the control PC: for the PC, the first crack appeared at the age of 17 days; for the MK5, MK10, and MK15 concretes, at 15, 10, and 12 days, respectively; for the SF5, SF10, and SF15 concretes, at 14, 9, and 7 days, respectively. In general, the onset cracking time and the stabilized crack width of the MK and SF concretes at the same replacement level were similar to each other, except for the replacement level of 15%.

Chloride diffusivity

Figure 9 shows that 15% MK had a significant improvement, while 5% MK had also some improvement on the chloride resistance. The 15% replacement of MK reduced the chloride concentration in Chamber B by approximately 55% after 43 days of diffusion. This result agrees well with the results from a previous study by Thomas, Gruber, and Hooton.[6] They demonstrated that the diffusion coefficient was reduced by approximately 50% for 8% MK and by approximately 60% for 12% MK. The SF concrete performed somewhat better than the MK concrete in increasing chloride resistance. After 90 days of standard curing, the effect of 10% SF was almost equal to that of 15% MK. At a replacement level of 15%, SF reduced the chloride concentration by approximately 73% compared with the control PC concrete after 43 days of diffusion, which was greater than the effect of 15% MK.

CONCLUSIONS

The effect of MK or SF on the workability, strength, shrinkage, and resistance to chloride penetration of concrete were investigated and compared in this study. For the given mixture proportions, MK offers better workability than does SF. As the replacement level was increased, the strength of the MK-modified concrete increased at all ages. The increase in the strength was similar to that of the SF-modified concrete. The incorporation of both MK and SF in concrete can reduce the free drying shrinkage and restrained shrinkage cracking width. The initial cracking appeared earlier in the SF- and MK-modified concrete, however, compared with the control PC concrete. The incorporation of MK or SF in concrete can reduce the chloride diffusion rate significantly, with the SF concrete performing somewhat better.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support of the Research Grant Council to the Hong Kong University of Science and Technology under Grant HKUST 6224/99E. The technical support from the Materials and Structural Laboratory of the Department of Civil Engineering is greatly appreciated.

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