Computers and Concrete, Vol. 10, No. 3 (2012) 219-229
DOI: http://dx.doi.org/10.12989/cac.2012.10.3.219

Research on chloride ion diffusivity of concrete subjected to CO_2 environment

Shiping Zhang* and Binghua Zhao

Department of Architecture Civil Engineering, Nanjing Institute of Technology,
Nanjing 211167, P.R. China

(Received November 29, 2011, Revised January 18, 2012, Accepted February 4, 2012)

Abstract. Carbonation is a widespread degradation of concrete and may be coupled with more severe degradations. An experimental investigation was carried out to study the effect of carbonation on chloride ion diffusion of concrete. The characteristic of concrete after carbonation was measured, such as carbonation depth, strength and pore structure. Results indicated that carbonation depth has a good linear relation with square root of carbonate time, and carbonation can improve compressive strength, but lower flexural strength. Results about pore structure of concrete before and after carbonation have shown that carbonation could cause a redistribution of the pore sizes and increase the proportion of small pores. It also can decrease porosities, most probable pore size and average pore diameters. Chloride ion diffusion of concrete after carbonation was studied through natural diffusion method and steady state migration testing method respectively. It is supposed that the chloride ion concentration of carbonation region is higher than that of the sound region because of the separation of fixed salts, and chloride ion diffusion coefficient was increased due to carbonation action evidently.

Keywords: concrete; carbonation; pore structure; chloride ion diffusion.

1. Introduction

Concrete is the most used construction material for infrastructures protecting the residential, activities and industrial spaces. Durability is a major concern for concrete structures exposed to aggressive environments. Many environmental phenomena are known to significantly influence the durability of reinforced concrete structures (Ihekwaba *et al.* 1996, Castro *et al.* 2000). Corrosion of steel reinforcement is one of the major causes of degradation of concrete structures, especially for sea-side structures, such as new airport, bridge and nuclear power plants. Carbonation and chloride attacks are two main factors of the corrosion of reinforcement in concrete structures (Yoon 2009). In normal circumstances when the carbon dioxide or chloride reaches the reinforced steel, the depassivation of the steel occurs and the steel reinforcement starts to corrode (Emmanuel and Ahmed 2009, Wanga 2005, Chindaprasirt *et al.* 2008). Furthermore, many studies have been done on the chloride attack in concrete structures (Wang and Ueda 2011, Mien *et al.* 2011). However, those studies were confined mostly to the single deterioration, although actual environment is rather of combined type.

Carbonation is one of the major factors to cause structure deterioration, and carbonation is much more widespread, because it involves carbon dioxide from air. Carbonation can be defined as the

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* Corresponding author, Ph.D., E-mail: zhangshiping1982@gmail.com

chemical reaction between carbon dioxide present in the air and cement hydration products such as mainly calcium hydroxide and the CSH gel phase, which results in the formation of calcium carbonate. Because of gas transport, carbon dioxide flows into concrete to react with hydration products, and oxygen flows through surface concrete to corrode steel reinforcement. Humidity and temperature conditions in this region may promote the advance of the carbonation front (Âleva *et al.* 1998, Castro *et al.* 1999). However, the actual carbonation rate will depend on several factors such as the type and amount of cement, porosity of the material, time of curing, type and quantity of pozzolanic additions, etc. Moreover, several modifications in concrete properties as compressive strength, superficial hardness and resistance to aggressive agents (e.g. sulfates) may occur due to carbonation (Castro *et al.* 2000). Carbonation of concrete itself does not do harm in view of the performance of structure, adversely a marginal enhancement of the compressive strength was observed. And the few reports published on the subject about investigation of other performance of concrete after carbonation action such as permeability are more or less contradictory. Carbonation reduces pH value and destroys the passive film around the steel, but it seems to densify concrete surface and reduce chloride ion permeability, reduce surface porosity and hence sorptivity in concrete (Dias 2000). Song also shown water permeability of hydrated mortars subjected to long term exposure to CO_2 can be decreased due to reduced porosity (Song and Kwon 2007). Reddy *et al.* found carbonation accelerates chloride ingress (Reddy *et al.* 2002), while Ihekwaba *et al.* shown that carbonation can reduce chloride ion diffusion into concrete (Ihekwaba *et al.* 1996). Glass *et al.* pointed out that the presence of even a small amount of chloride in carbonated concrete enhances the corrosion rate resulted from carbonation of concrete (Glass *et al.* 1991). Tumidajski *et al.* also found that CO_2 can decrease the chloride penetration and diffisivity in the ordinary Portland cement concrete (Tumidajski and Chan 1996).

Carbonation could have both positive and negative effects on concrete durability. This paper describes an investigation into chloride ion diffusion of concrete subjected to carbonation. The experimental study includes mercury intrusion porosimetry on concrete, accelerated carbonation test and chloride ion diffusion testing, in order to study the effect of carbonation action on chloride ion diffusion of concrete.

2. Experimental program

2.1 Raw materials

Samples were prepared with ordinary Portland cement (OPC) produced by Onada Corp. in Jiangsu Province, China. Chemical composition of the cement is presented in Table 1. Crushed stone with a maximum size of 25 mm, quartz sand with a fineness modulus of 2.3 were used. Concrete admixture used was JM-PCA produced by Jiangsu Bote New Materials Co., Ltd.

Table 1 Chemical compositions of cement

Sample	Chemical composition/*wt*%						
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2Oeq
Cement	66.6	21.74	5.06	3.56	1.60	0.81	0.50

2.2 Experimental method

The concrete prisms with size of 100 mm × 100 mm × 100 mm and 100 mm × 100 mm × 400 mm were cast, and samples size of 100 mm × 100 mm × 400 mm were specially used for flexural strength testing (using 3 point bend test). Strength testing was carried out according to Chinese National Standard GB/T 50081-2002. The mix proportions of concrete are listed in Table 2. After 24 h, the experimental specimens were demoulded and standard cured at 20°C±3°C, humidity was controlled at 90±5% for 28 days. Then they were cut into 50 mm thick slices with the 50 mm ends discarded. The samples were separated into two groups namely group A and group B. The group A samples were used for the strength testing, natural diffusion testing and steady state migration testing, and the group B samples were subjected to accelerated carbonation for different period and were then tested for the strength testing, natural diffusion testing and steady state migration testing.

Since carbonation reactions are so slow in natural environments, accelerated methods are necessary for measurement of non-time dependent relationships in reasonably short time frames. Carbonation testing was carried out according to Chinese National Standard GBJ82-1985. The main result of the accelerated carbonation test is carbonated depth. This was assessed by a colorimetric method based on the drop of pH at the carbonation front. Temperature was maintained at room temperature, 20°C±3°C, and humidity was controlled at 70±5%. The concentration of carbon dioxide was 20±3%. After carbonation testing, samples were broken perpendicular to the surface, and then spray the freshly broken surface with a 1-2% phenolphthalein solution. The phenolphthalein solution leaves the surface clear when the pH is less than 9 and turns magenta when the pH is above 9. This method allows measurement of the penetration of complete carbonation by change in color. Carbonation depth was measured after certain time. Carbonated specimens were measured at 7, 14, 28 and 56 days, and the specimen unbroken from the same batch was used for chloride ion diffusion testing.

Several test methods exist for testing of the chloride migration in concrete. Natural diffusion method and steady state migration testing method were chosen. For steady state migration testing method, the driving force is an electrical potential that forces ions to migrate between the anode and the cathode. The schematic diagram of steady state migration testing is shown in Fig. 1. This migration test consists in accelerating a chloride ion diffusion test by a constant electrical field. The

Table 2 Mix proportion of concrete

Cement/kg/m^3	Water/kg/m^3	Sand/kg/m^3	Crushed stone/kg/m^3	Admixture/kg/m^3	Air content/vol.%
440	176	665	1235	1.368	3.3

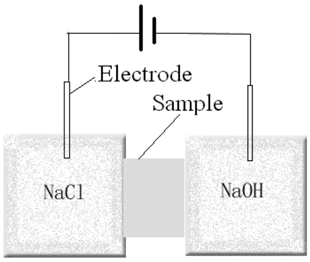

Fig. 1 Schematic diagram of steady state migration testing

effective chloride ion diffusion coefficient is assessed from the chloride ion flux in the cathodic cell in steady state. The 50 mm slices were prepared including vacuuming, saturation and setting up the test, and the lateral surface of specimens was coated with epoxy, then placed between two cells. Each specimen was placed between the two compartments of a cell where flat silicone circular seals ensure that the system is leaktight. The solutions were made with NaCl (3%) in the upstream compartment, and NaOH (0.2 mol/l) in the downstream compartment. A 8 volt potential was applied over the specimen, and the test was carried out at temperature $T = 20 \pm 5$°C. Electrical current was simultaneously monitored in the migration test, and finally chloride-ion concentration was measured. This migration testing consists in accelerating a chloride diffusion testing by a constant electric field. The effective chloride diffusion coefficient is assessed from the chloride ion flux in the cathodic cell in steady state.

Besides, natural diffusion method was also used to evaluate chloride ion diffusion of carbonated concrete. 50 mm slices with carbonation and without carbonation were chosed for natural diffusion method. The side and bottom of the specimens were coated with epoxy leaving only top face uncoated after dried. The specimens were immersed in 5% NaCl solution for 60 days in a controlled room at 20°C±3°C. A drilling machine with a bit that can be adjusted to different sizes was used to take samples. The bit was moved at regular intervals. The sampling diameter was 6 mm. After each milling step, the resulting powder was collected and then was used to extract acid-soluble chloride contents. Mohr titration was used to determine the chloride concentration in the solution. This method allows us to determine the profile of the chloride concentration as a function of depth.

3. Results and discussion

3.1 Performance of carbonated specimens

Carbonation depth of concrete samples after 7, 14, 28 and 56 days was measured. Observations of the fresh broken surfaces, on which phenolphthalein was sprayed, show a clear border line between

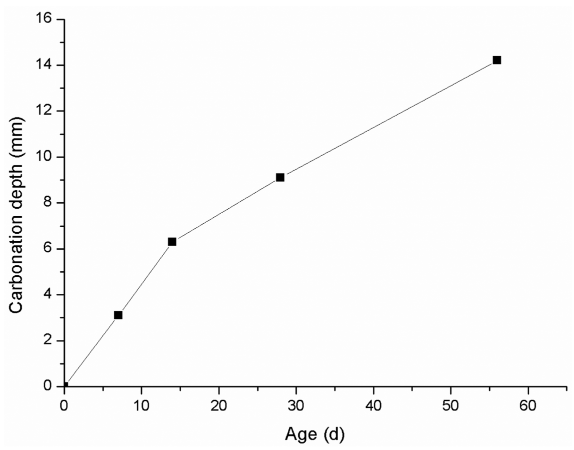

Fig. 2 Carbonation depth of concrete samples

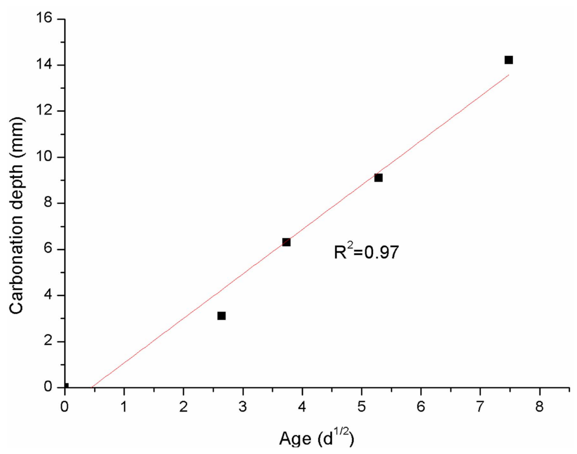

Fig. 3 Relations between carbonate depth and square root of carbonate time

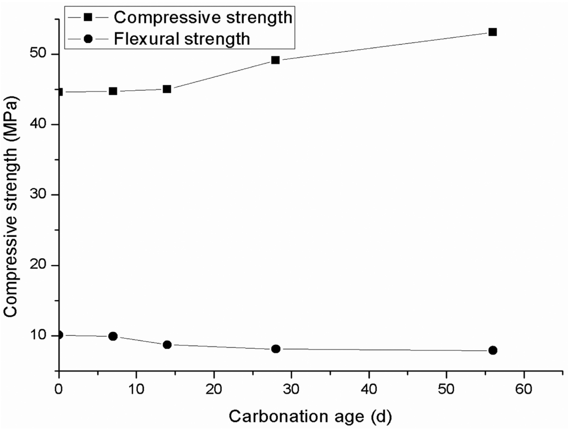

Fig. 4 Compressive strength and flexural strength of concrete

red and unchanged surfaces. Fig. 2 shows the results of carbonation depth of concrete samples. From the Fig. 2, it can be seen that longer carbonate time leads to higher carbonate contents and increases carbonation depth. Furthermore, according to experimental data, carbonation depth has a good linear relation with square root of carbonate time, as shown in Fig. 3 (Ruan and Pan 2012).

Compressive strength and porosity may be regarded as general indicators for potential durability. So in order to better understand results from measures of global properties, strength testing and mercury intrusion porosimetry (MIP) testing have also been carried out. Fig. 4 gives results of compressive strength and flexural strength of concrete before and after carbonation. According to Fig. 4, it can be seen that compressive strength of carbonated concrete was greater than that of noncarbonated concrete, and compressive strength was gradually increasing with carbonation age. However, carbonation decreased flexural strength, and the longer carbonation age caused serious loss of flexural strength. So, it indicated that carbonation can improve compressive strength, but lower flexural strength, which will affect the toughness and cracking resistance of concrete materials.

The pore structure of cement-based materials is one of its most important characteristics and strongly influences both its mechanical behavior and its transport properties. The durability of cement-based materials depends on their transport properties, and it is affected by the pore structure (Yang 2006). Pore size distribution curves and quantitative results, such as porosities, median and average pore diameters may be useful to analyze properties of concrete (Roy *et al.* 1999, Villain and Platret 2006). Cement-based materials contain air voids, capillary pores and gel pores (the interlayer spaces in a C-S-H gel), and the pores in concrete are randomly sized, arranged and connected (Samaha and Hover 1992). Because of the wide range of feature sizes in pore, from nanometer-sized pores to micrometer-sized air voids (Mehta and Monteiro 1993), the microstructure of cement paste is very complex. The mercury intrusion porosimetry (MIP) is the most widely used method for the study of pore structure characteristics of cement-based materials. The MIP method has also been widely used to investigate the pore structure of cement and concrete materials (Ngala e*t al.* 1995, Uchikawa *et al.* 1996, Shi 1996, Jiang and Guan 1999, Pandey and Sharma 2000, Frias and Cabrera 2000, Matusinovic *et al.* 2003, Ngala and Page 1997). Mercury intrusion porosimetry (MIP) (GT-60, made by Quantachrome Co., USA) was used here. MIP tests were carried out on concrete samples after 56*d* carbonation and noncarbonated sample. Results about pore size distribution of

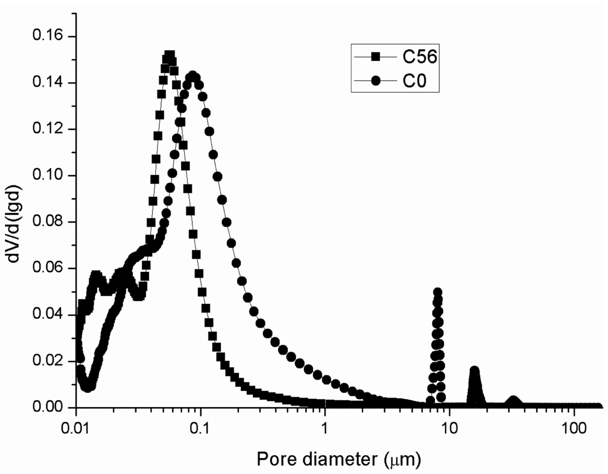

Fig. 5 Pore size distribution of concrete specimens

Table 3 Pore structure parameters of concrete specimens

Sample	Total porosity /%	Average pore diameter/nm	$d \geq 200$ nm /%	$40 \leq d \leq 200$ nm/%	$d \leq 40$ nm/%
C56	16.19	52.06	4.14	61.46	34.40
C0	21.91	91.88	19.16	60.87	19.97

carbonated and non-carbonated samples are shown in Fig. 5. *C*56 represented the sample after 56*d* carbonation, while *C*0 represented the sample without carbonation. According to Fig. 5, it can be deduced that carbonation affects pore size distribution of concrete, and most probable pore size becomes small. Detail data about pore structure carbonated and non-carbonated samples is listed in Table 3. From the Table 3, it is found that about 26% decrease in porosity and reduction in the average pore size were observed between the carbonated and noncarbonated samples. For pore size distribution, the proportion of small pores (diameter ≤ 40 nm) was increased, and the proportion of large pores (diameter ≥ 200 nm) was reduced. As we known, during carbonation process, carbon dioxide reacts with calcium hydroxide to form calcium carbonate, there is a solid volume increase of around 11% in the conversion of $Ca(OH)_2$ to $CaCO_3$ (Song and Kwon 2007). This involves an expansion of solid volume in the paste and hence a reduction of porosity. So, carbonation could cause a redistribution of the pore sizes and increase the proportion of small pores.

3.2 Chloride ion diffusion

Chloride binding has an influence upon time lag but it does not alter the value of the diffusion coefficient (David *et al.* 2010). Therefore, we are able to determine the diffusion coefficient of concrete within a short period by using the migration test. In chloride migration testing, chloride ions are transported in concrete under an applied voltage. As the chloride flux becomes constant, the general equation that describes transport processes in solution is the Nernst-Planck's equation (Misra *et al.* 1994)

$$J = -D\frac{\partial C}{\partial x} - \frac{zF}{RT}DC\frac{\partial V}{\partial x} + Cu \tag{4}$$

where D is the diffusion coefficient of ion (m^2/s), which is later defined as a migration coefficient of mortar; T is the concentration of ion (mol/m^3); R is the universal gas constant (8.3 *J*/mol/K); T is absolute temperature (*K*); *V/x* is the strength of the electric field between the anode and cathode (V/m) and u is the velocity of solute (m/s). The first and the third terms of Eq. (4) are the contribution due to diffusion and convection, respectively. Since the concrete is saturated, the velocity of solute can be neglected. Under the influence of an electrical field across the sample, the contribution of diffusion in concrete is small and can be neglected (Yang *et al.* (2003)). Then, only taking into account the migration due to the electrical field, the migration coefficient D can be calculated by rearranging the middle term of Eq. (4)

$$J = -\frac{zF}{RT}DC\frac{\partial V}{\partial x} \tag{5}$$

Then the migration coefficient of chloride ion for concrete, D_{cl}, is calculated as

$$D_{cl} = \frac{RT}{zFC(E/l)}J_{cl} \tag{6}$$

where J_{cl} is constant flux of chloride in the downstream cell (mol m^{-2} s^{-1}), z is the electrical charge of chloride, F the Faraday constant (96,500*C*mol^{-1}), and C the chloride concentration in the upstream cell at the cathode

$$J_{cl} = \frac{V}{A}\frac{\partial C}{\partial t} \tag{7}$$

From Eqs. (4) and (7), the migration coefficient of chloride ion of concrete is calculated from a stable steady-state current as follows (Yang *et al.* 2002)

$$D_{cl} = \frac{RTt_{cl}}{zCF^2A(E/l)}I_s \tag{8}$$

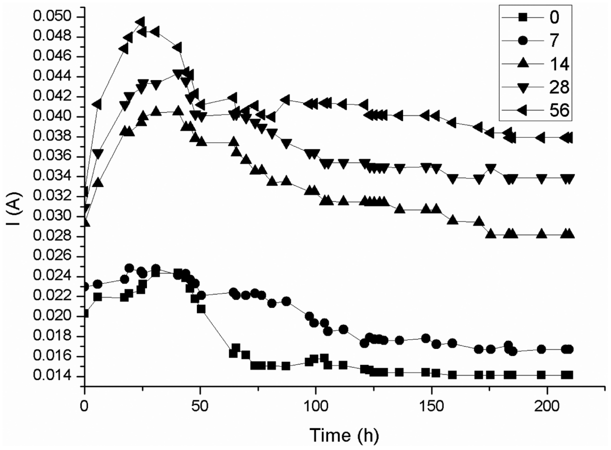

Fig. 6 Electric current during chloride migration testing

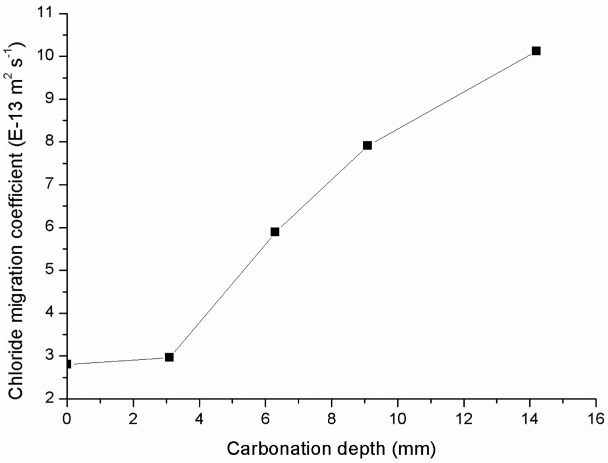

Fig. 7 Chloride migration coefficient of samples

Electrical current was simultaneously monitored in the migration test, and the electrical current results of different carbonation age were shown in Fig. 6. The chloride migration coefficients of concrete samples calculated from Eq. (8) are also shown in Fig. 7. It is apparent that the chloride migration coefficient of carbonated sample is greater than that of noncarbonated sample. Furthermore, the higher the carbonation depth was, the greater the chloride migration coefficient. It is well known that permeability is depended on porosity, and the concrete of low porosity often shows well impermeability. However, results about chloride migration coefficients of carbonated concrete are not consistent with that theory, based on the reduction of porosity due to carbonation.

In order to study the chloride ion diffusion of carbonated concrete in further, the immersion test of concrete was performed. The positions of sampling for chloride profiling on the specimen are shown in Fig. 8. All the concentration data presented here are in percent weight of concrete which resulted from the chemical analysis test. The concentration value at a particular depth means an average concentration within 2-mm depth. The chloride ion concentration profiles of concrete after

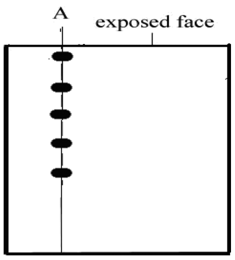

Fig. 8 Instruction of testing sites for chloride profiling

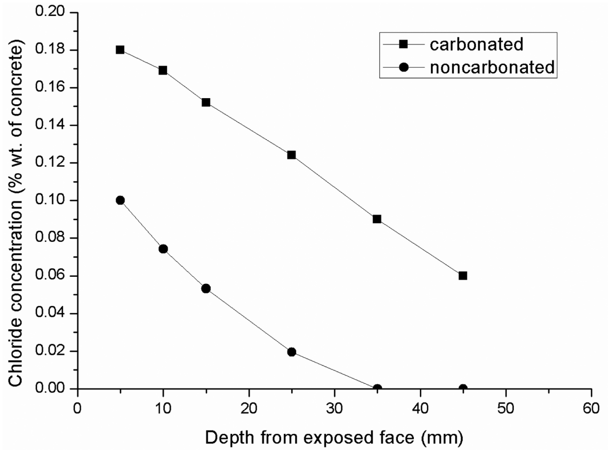

Fig. 9 Chloride ion concentration of concrete samples

56*d* carbonation and noncarbonated concrete are shown in Fig. 9. As expected, from the surface to interior, a clear trend is found that the chloride ion concentration is high near the surface. According to Fig. 8, the largest amount of chloride ion penetrating in concrete and the corresponding variations according to depth were observed for carbonated concrete. Besides, the chloride content of carbonated concrete appears to be larger at depth of 5 mm from the surface than that of noncarbonated concrete, and the chloride content for carbonated concrete appears to be significantly larger than that of noncarbonated concrete at any position. Carbonation can alter the pore structure and the permeability of concrete. The binding capacity for aggressive ions is also affected due to carbonation. Chemically combined and physically adsorbed chloride ions are set free with the advance of carbonation, increasing the number of free chloride ions in concrete (Basheer *et al.* 2001). For example, free chloride fixed as Friedel's salt in the cement hydrates would be released. Carbonation lowers the pH and thus affects the disassociation of the bound chlorides in the form of Friedel's salt. The solubility of Friedel's salt increases with the degree of atmospheric carbonation of the concrete. Hence, carbonation chemically increases the chloride content (Suryavanshi and Swamy 1996). This is the likely cause of the high chloride content at carbonation area near surface. Besides, for carbonated concrete, when the concentration of free chloride ions increases in the pore solution of the carbonated area according to the progress of carbonation, the gradient of the concentration of free chloride ions increases at the interface between the carbonated and non-carbonated zones, therefore contributes to the migration of the chloride ions toward the non-carbonated zone. This migration results finally result in larger chloride content in the non-carbonated zone. So, chloride ion can penetrate deeper, compared with noncarbonated concrete. Then chloride ion diffusion of carbonated concrete is greater than that of noncarbonated concrete.

4. Conclusions

Based on the experimental work and the analysis of the test results, the following conclusions can be drawn: Longer carbonate time leads to higher carbonation depth, and carbonation depth has a good linear relation with square root of carbonate time. Carbonation also can improve compressive strength, but lower flexural strength, which will affect the toughness and cracking resistance of concrete materials. Carbonation could cause a redistribution of the pore sizes and increase the proportion of small pores. It also can decrease porosities, most probable pore size and average pore diameters. Carbonation can accelerate chloride ion diffusion in concrete, and chloride ion diffusion coefficient was increased due to carbonation action. This can be explained by the growing diffusivity of chloride ion provoked by increased concentration gradient of chloride ion due to the release of Friedel's salt in the carbonated zone. Furthermore, the concentration gradient of free chloride ions increases at the interface between the carbonated and non-carbonated zones, which contributes to further migration of chloride ions toward the non-carbonated zone.

Acknowledgements

This research was supported by National Natural Science Foundation of China (Grant No. 51108231), the Natural Science Foundation of Jiangsu Province (Grant No. BK2011690) and State Key Laboratory of High Performance Civil Engineering Materials (Grant No. 2010CEM019).

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