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A new chemo-mechanical model of damage in concrete under sulfate attack



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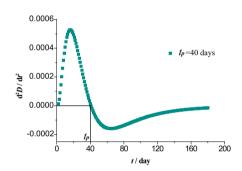
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HIGHLIGHTS

- Damage evolution in concrete under sulfate attack is derived considering chemical reaction rate.
- The characteristic time of chemical reaction represents inflection point of damage evolution.
- Damage nucleation is due to tensile failure at the surface of pores by delayed ettringite.

G R A P H I C A L A B S T R A C T

The characteristic time t_p related chemical reaction rate approximately is the inflection point of damage evolution.



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ABSTRACT

Damage evolution is detected via the degradation of modulus of concrete immersed in sulfate solution. The mechanism of the damage is investigated by using SEM method, and it is proved that the internal expansion stress induced by delayed ettringite leads to the nucleation of micro-cracks. The criterion of damage nucleation is proposed in terms of the tension strength of concrete. The differential equation with respect to the internal expansion stress is derived by virtue of chemical reaction rate on delayed ettringite formation, and a new chemo-mechanical model of corrosion damage in concrete under sulfate attack is then proposed.

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1. Introduction

In the environment of ocean or salt lake, the mechanical properties of concrete structures strongly degrade by attack of harmful ions, such as chloride ions, sulfate ions, magnesian ions, and so on

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[1,2]. It is well known that chloride ions dominantly attack reinforcing steel and induce it to corrosion, and magnesian ions react with C-S-H gel, and lead to calcium leaching. The mechanism for the sulfate attack on concrete matrix, however, is so complicated that it is still unclear till now. For instance, Tixier and Mobasher [3] and Ikumi et al. [4] thought that a series of chemical reactions take place between sulfate ions and the unreacted hydration compounds of the cement paste, and all those reactions lead to degradation of concrete. Therefore, this challenge problem attracts many

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researchers' close attention. In fact, people early realized the degradation effect of sulfate in the second half of the nineteenth century, and found sulfate ions diffused in porous of concrete react with pore solution to induce delayed ettringite formation (DEF). DEF leads to the damage evolution under the condition that concrete dose not experience an elevated temperature (e.g. [5,6]). Related studies on concrete under sulfate attack dominantly focus on the mechanism of degradation of mechanical properties and material design on sulfate resistance.

Because the degradation mechanism of mechanical properties relates to the delayed ettringite, some of studies focus on the mechanism of DEF [7,8] and crystal structure [9,10]. The internal expansion forces are produced under the action of delayed ettringite, and the expansion in concrete occurs due to the internal expansion forces. Barbarulo et al. [11] studied the expansion of concrete during DEF under high temperature and thermal cycle, and found that the expansion strain reaches 3%. Payoine et al. [12] further considered the effect of wetting and drying on the expansion of concrete due to DEF, and indicated that the expansion occurs earlier than that in the concrete without wetting and drying condition. Zhang et al. [13] studied the expansion of concrete due to attack of sulfate and sulfate-chloride ions. It was found that the expansion caused by attack of sulfate-chloride ions is much less than that induced only by sulfate attack. The mechanism of such a phenomenon is that DEF in the former situation is much less than that in the latter situation.

Under sulfate attack, the corrosion damage leads to the degradation of stiffness and strength of concrete. Planel et al. [14] studied the long-term behavior of cement mortar, and analyzed size-effect on the degradation of mechanical properties of the material. Chen et al. [15] investigated the damage evolution in concrete due to sulfate attack, and suggested an empirical formula of the damage evolution. Rozière et al. [16] not only experimentally analyzed the variation of mass and deformation of concrete exposed to leaching and external sulfate attacks, but also showed some pictures of cracks growth in micro-scale. Because the filling effect of delayed ettringite in pores of concrete, the pore structure is changed. Pipilikaki et al. [17] examined the variation of microstructure of concrete under sulfate attack, meanwhile they also inspected the changes of compressive strength of the material.

In order to improve the property of sulfate resistance of concrete, some researchers adopted new mix design of concrete. For instance, Plowman and Cabrera [18] used fly ash as additive agent to polish up the microstructure of concrete; Chindaprasirt et al. [19] employed both fly ash and rice husk ash as new components of concrete for slowing down degradation of concrete; Lee et al. [20] replaced part of cement by silica fume to reduce the magnitude of delayed ettringite in concrete.

All those method can only slow down the rate of chemical reaction for DEF, however, it is difficult to completely prevent concrete from degradation. Therefore, it is still important to establish a reasonable prediction theory on the service life of concrete structure in marine environment. Kuhl et al. [21,22] suggested a new theoretical frame, which includes the coupling effect of chemistry and mechanics, to analyze the degradation of cementitious materials due to calcium leaching and mechanical damage. Nevertheless, few works on the degradation of concrete under sulfate attack have been reported.

In this paper, variation of dynamic modulus of concrete under sulfate attack was detected by virtue of ultrasonic technique. The average corrosion damage was defined as the relative decrease to the modulus, and it was determined from the experimental results. Then, the damage nucleation due to the action of internal expansion stress, which is produced by delayed ettringite, was analyzed by Eshelby's equivalent inclusion method. Based on the assumption that the expansion stress is directly proportional to the mass

of delayed ettringite, the differential equation for the expansion stress is derived by considering the related chemical reaction as well as reaction rate. Finally, a new damage evolution model of concrete is proposed in terms of Weibull's weakest link model.

2. Experimental

Reference cement produced by China Building Materials Academy was adopted to form specimens of concrete. The components of the reference cement were listed in Table 1.

Cement-to-sand ratio of the specimens is 1:2, and water-to-cement ratio is 0.45. The shape of the specimens is cylindrical, which are 500 mm in length and 45 mm in diameter, respectively. The concrete specimens were cured 24 h at ambient condition, after then they were put into a concrete standard compartment for curing 28 days, which has the environment temperature of $20\,^{\circ}\mathrm{C}$ and relative humidity of 90%.

After being cured 28 days, the specimens were moved into the solutions of sodium sulfate, with three different concentrations selected as, 3%, 5%, and 8%. During the period of immersion, the ultrasonic detection was carried out, and diagram of the detection was plotted in Fig. 1. When the frequency of ultrasonic is given, the wave propagates from one end of the specimen to the other end with a constant velocity.

In the situation shown in Fig. 1, the ultrasonic propagation is an axisymmetric problem. Just as pointed by Rayleigh, however, if the ratio of radius to wavelength is less 0.7, then it can be approximately referred to as one dimensional stress [23]. The relation between the elastic modulus, *E*, and the wave velocity, *C*, can be written as follows [24],

$$E = C^2 \rho \tag{1}$$

where the symbol " ρ " stands for density of the specimen. If the change of the density and the wave velocity are measured, then the variation of the modulus can be determined by Eq. (1).

By means of weight method, variation of density was measured at different immersion times and plotted in Fig. 2. It can be seen that the density monotonously increases due to a series chemical reactions, including reactions on continued hydration and DEF, etc.

Variation of modulus was measured by virtue of ultrasonic technique shown in Fig. 1, and the results were plotted in Fig. 3. The time interval of the first stage decreases with the increase of the concentration of sulfate solution. It was found that the variation of the modulus can be divided into two stages. In the first stage (initial stage) the modulus increases, but in the second stage it decreases. This implies that the damage evolution only takes place in the second stage, thus, we define that the damage in this stage is the relative decrease of the modulus, i.e.,

$$D = 1 - \frac{E}{E_{\text{max}}} \tag{2}$$

where the symbol " $E_{\rm max}$ " denotes the maximum modulus. From the experimental results in Fig. 3 and Eq. (2), the damage was obtained and plotted in Fig. 4. One can see that damage evolution is more fast in the sulfate solution of higher concentration.

After being immersed for 360 days, the macro-cracks can be observed in specimens as shown in Fig. 5.

For establishing a theoretical model of damage evolution, microscopic observation was performed using SEM technique. Fig. 6 shows the observation of DEF in pores of concrete and related energy spectrum analysis.

3. Theoretical model of damage evolution

Damage evolution in concrete can be studied by investigating the crack propagation [25], or predicting statistical evolution of damage by virtue of continuum damage [26]. In this study, a statistic method is adopted to investigate the damage evolution in concrete under sulfate attack. For this, the damage nucleation is studied first.

3.1. Damage nucleation

Experimental results indicate that DEF in pores of concrete is a dominant factor to induce the corrosion damage. The mechanism for damage nucleation is that sulfate ions diffused in pores of concrete react with pore solution in concrete, which leads to DEF (as shown in Fig. 7). This process can be divided into two steps: 1) reaction between sulfate ions, SO_4^{2-} , and calcium hydroxide, CH, to form gypsum, $C\bar{S}H_2$; 2) reaction of gypsum with tricalcium

Table 1 Components of reference cement.

SiO ₂	Al_2O_3	Fe_2O_3	CaO	MgO	SO ₃	Na_2O_{eq}	LOSS	f-CaO	C3S	СЗА
21.07	4.45	2.69	63.23	2.03	2.93	0.53	2.38	0.94	53.01	6.68

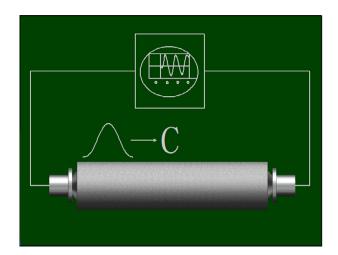


Fig. 1. Diagram of ultrasonic detection.

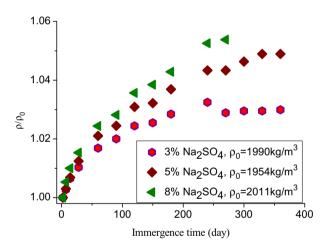


Fig. 2. Evolution of density of concrete under sulfate attack.

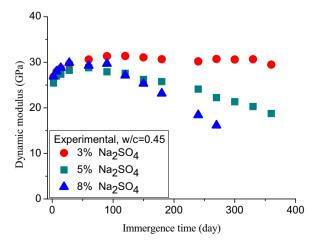


Fig. 3. Evolution of modulus of concrete under sulfate attack.

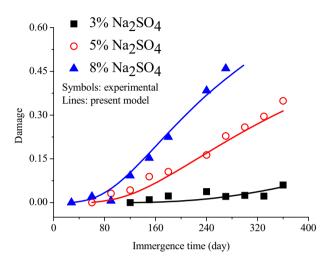


Fig. 4. Damage evolution in specimens under sulfate attack.



Fig. 5. Macro-cracks in specimens under sulfate attack.

aluminate, C_3A , and water, H, to form delayed ettringite, $C_6A\bar{S}_3H_{32}$. Detail chemistry reaction equations are given as follows [4],

$$CH + SO_4^{2-} \longrightarrow C\bar{S}H_2 + 2OH^- \tag{3}$$

$$\begin{split} \text{CA} + q \bar{\text{CSH}}_2 &\longrightarrow \text{C}_6 A \bar{\text{S}}_3 \text{H}_{32} \\ \text{CA} := \gamma_1 \text{C}_3 \text{A} + \gamma_2 \text{C}_4 \text{AH}_{13} + \gamma_3 \text{C}_4 A \bar{\text{S}} \text{H}_{12} \\ q = 3\gamma_1 + 3\gamma_2 + 2\gamma_3 \end{split} \tag{4}$$

where symbols " C_4AH_{13} " and " $C_4A\bar{S}H_{12}$ " respectively represent tetracalcium aluminate and monosulfate; q stands for the stoichiometric weighted coefficient of the sulfate phase; γ_i (i = 1–3) denotes the proportion of each aluminate phase to the total aluminate content.

As shown in Fig. 7, if a pore is filled by the delayed ettringite, expansion stress, *p*, begins to appear, and it produces tensile stress at the surface of the pore. When such a tensile stress reaches the

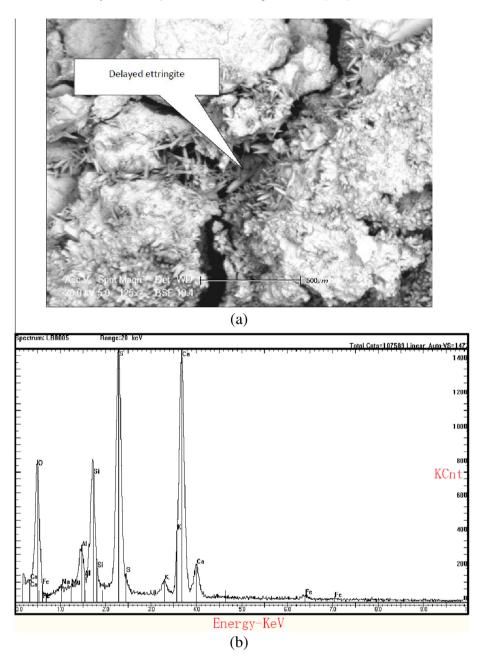


Fig. 6. Delayed ettringite formation: (a) SEM photo; (b) energy spectrum analysis.

tensile strength of the material, micro-cracks appear, and this is the mechanism of the nucleation of micro-damage. Here, we use the symbol " $p_{\rm cr}$ " to denote the critical expansion stress when the nucleation of micro-damage appears. In the following analysis, the Eshelby's equivalent inclusion method [27] is adopted to evaluate $p_{\rm cr}$.

For convenience, the shape of a pore is approximately assumed to be ellipsoidal void of revolution (shown in Fig. 8), in which the semi-axes of the pore in x-, y-, and z-directions are denoted by a_1 , a_2 , and a_3 (a_1 = a_2), respectively. Usually, the viscous effect of concrete should be taken into account for establishing the constitutive model. In this study, however, the nucleation of micro-cracks is induced by tensile force, and in this situation, the deformation is so small that the constitutive relation of concrete can be approximately assumed to be a linear isotropic elastic one, i.e.,

$$\begin{aligned} & \boldsymbol{\sigma} = \boldsymbol{L} : \boldsymbol{\epsilon} \\ & \boldsymbol{L} = 3k\boldsymbol{I}_m + 2\mu\boldsymbol{I}_s \end{aligned} \tag{5}$$

where k and μ are the bulk and shear moduli, respectively, and $I_{\rm m}$ and $I_{\rm s}$ are the fourth order tensors defined by Chen et al. [28],

$$\mathbf{I}_{\mathbf{m}} = \mathbf{i}_{\mathbf{0}} \otimes \mathbf{i}_{\mathbf{0}}/3, \quad \mathbf{I}_{\mathbf{s}} = \mathbf{I} - \mathbf{I}_{\mathbf{m}} \tag{6}$$

where ${\bf I}$ and ${\bf i}_0$ are the fourth order unit tensor and the second order unit tensor, respectively.

In terms of Eshelby's equivalent inclusion method, the relation between the pore strain, ϵ_p , and the remote strain, ϵ^{∞} , can be expressed as [29]

$$\boldsymbol{\varepsilon}_{p} - \boldsymbol{\varepsilon}^{\infty} = \mathbf{S} : (\boldsymbol{\varepsilon}_{p} + p\mathbf{J} : \mathbf{i}_{0})$$
 (7)

where ${\bf S}$ is the fourth order Eshelby's tensor; ${\bf J}$ is the compliance tensor, and

$$\mathbf{J} = \mathbf{L}^{-1} = \mathbf{I}_{\rm m}/(3k) + \mathbf{I}_{\rm s}/(2\mu) \tag{8}$$

If there is no external loads, then the remote strain is zero, the relation between ϵ_p and p can be expressed by solving Eq. (7) as follows.

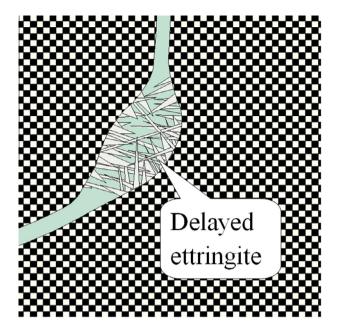


Fig. 7. Plot of delayed ettringite formation in a pore of concrete.

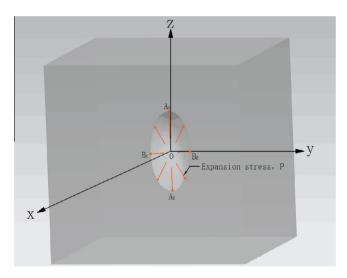


Fig. 8. Plot of a void with expansion stress.

$$\begin{aligned} & \boldsymbol{\epsilon}_p = p \boldsymbol{g} \\ & \boldsymbol{g} = \left(\boldsymbol{I} - \boldsymbol{S} \right)^{-1} : \boldsymbol{S} : \boldsymbol{J} : \boldsymbol{i}_0 \end{aligned} \tag{9}$$

Let us now calculate the stress at A_1 or A_2 , which are the two poles of the void. It is obvious that the stress at point A_1 is the same as that at A_2 due to symmetry. Therefore, only stress components at A_1 are calculated in the following analysis.

According to the boundary condition at the surface and symmetrical property with respect to z axis, i.e.,

$$\sigma_{33}|_{A1} = -p \tag{10}$$

$$\sigma_{11}|_{A1} = \sigma_{22}|_{A1}, \quad \varepsilon_{11}|_{A1} = \varepsilon_{22}|_{A1}$$
 (11)

It is easy to obtain the following results,

$$\sigma_{11|A_1} = \beta p \beta = (6\lambda \mu g_{11} + 4\mu^2 g_{11} - \lambda)/(\lambda + 2\mu)$$
(12)

where g_{11} can be calculated from Eq. (9).

If $\sigma_{11}|_{A1}$ reaches the tensile strength, σ_0 , the damage nucleation occurs, and at this instance, p reaches the critical expansion stress, p_{CP} . Therefore, we obtain,

$$p_{\rm cr} = \beta^{-1} \sigma_0 \tag{13}$$

Eq. (13) gives the relation between the critical expansion stress and the tensile strength of concrete. If the Poisson's ratio v = 0.18, and the shape of the pore is spherical, then $\beta = 0.159$.

3.2. Evolution of expansive stress

It can be realized that the development of expansion stress depends on the increase speed of delayed ettringite, which relates to the rate of chemistry reaction. The concentration of SO_4^{2-} and C_3A is denoted by symbols " C_{SO} " and " C_{CA} ", respectively, then the chemical reaction rate equation for Eqs. (3) and (4) is given by Tixier and Mobasher [3],

$$\frac{dC_{CA}}{dt} = -\frac{kC_{CA}C_{SO}}{q}$$

$$\frac{dC_{SO}}{dt} = -kC_{CA}C_{SO}$$
(14)

where k is the coefficient of reaction. Form Eq. (14) we obtain, Solving Eq. (14) we obtain,

$$C_{SO} = qC_{CA} \tag{15}$$

Substituting Eq. (15) into Eq. (14) yields,

$$\frac{\mathrm{d}C_{\mathrm{CA}}}{\mathrm{d}t} = -kC_{\mathrm{CA}}^{2} \tag{16}$$

It is well known that expansive stress depends on the amount of delayed ettringite, for simplicity, it is approximately assumed that the increment increase of the expansive stress, dp, is directly proportional to the increase of concentration of delayed ettringite, $dC_{ettringite}$, i.e.,

$$dp \propto dC_{\text{ettringite}}$$
 (17)

Eq. (4) indicates that the increase of concentration of delayed ettringite is directly proportional to the decrease of concentration, dC_{CA} , i.e.,

$$dC_{ettringite} \propto -dC_{CA}$$
 (18)

From Eqs. (17) and (18) we obtain,

$$dC_{CA} = -\lambda dp \tag{19a}$$

or

$$\mathsf{C}_{\mathsf{CA}} = -\lambda(p - p^*) \tag{19b}$$

where λ and p^* are the parameters to be determined. Substituting Eq. (19) into Eq. (16) we obtain,

$$\frac{d\mathbf{p}}{d\mathbf{t}} = k\lambda (p - p^*)^2 \tag{20}$$

Eq. (20) is the evolution equation of expansive stress. Solving Eq. (20) for p we obtain,

$$p = p^* \frac{t/t_p}{1 + t/t_p}$$

$$t_p = 1/(k\lambda)$$
(21)

where t_p is a characteristic time to be determined, which is related to the coefficient of the reaction rate, k. Eq. (21) gives an evolution function of expansive stress.

3.3. Model of damage evolution

According to the previous analysis, the damage evolution of concrete under sulfate attack is induced by tensile stress at the surface of pores. Under tensile loading, concrete possesses brittle characteristic, hence, damage evolution can be described by weakest-link theory [30,31]. Therefore, we assume that the damage evolution obeys Weibull's distribution function, i.e.,

$$D = 1 - \exp\left[-\left(\frac{p - p_{cr}}{p_u}\right)^m\right], \quad (p \geqslant p_{cr})$$
 (22)

where m and p_u are the parameters to be determined, and p_{cr} is determined by Eq. (13).

Substituting Eqs. (21), (22) yields,

$$D = 1 - \exp \left[-\left(\frac{p^* \frac{t/t_p}{1 + t/t_p} - p_{cr}}{p_u} \right)^m \right]$$
 (23)

If the damage nucleation occurs at a critical instant t_{cr} , then from Eq. (23) we obtain,

$$p_{cr} = p^* \frac{t_{cr}/t_p}{1 + t_{cr}/t_p} \tag{24}$$

The critical nucleation time, t_{cr} , is directly determined by experimental results shown in Fig. 4, which is the initial instant for damage evolution. The values of t_{cr} are listed in Table 2.

Substituting Eq. (24) into Eq. (23), we obtain a new model of damage evolution as follows,

$$D = 1 - \exp\left\{-\left[\gamma\left(\frac{t/t_p}{1 + t/t_p} - \frac{t_{cr}/t_p}{1 + t_{cr}/t_p}\right)\right]^m\right\}, \quad (t \ge t_{cr})$$

$$\gamma = \frac{p^*}{p_u}$$
(25)

There are only three parameters in Eq. (25), γ , t_p , and m, that need be determined. The rate of reaction is represented by the characteristic time t_p . All parameters are determined according to the experimental results by using a curve fitting method, the values of which are listed in Table 2.

4. Discussion

Most researches on the problems of concrete under sulfate attack dominantly attributed the degradation mechanism to the action of delayed ettringite, which was observed in micro-cracks. Perhaps some people argue that such micro-cracks are induced by other factors, such as carbonation, drying and wetting cycle, freezing and thawing cycle, attack of chloride ions, or original defects in concrete, and those defects supply space for delayed ettringite formation and growth. Hence, they consider that delayed ettringite dose not cause such a degradation of concrete. In this study, the experiment design excludes those factors. For instance, specimens are formed by cement mortar, and there is no aggregate in the material (for reducing the original defects), and the specimens were immersed in sulfate solution at room temperature

Table 2 Parameters of damage model.

Parameters/concentration (%)	t _{cr} (day)	γ	m	t _p (day)
3	120	1.5	4.236	350
5	60	1.5	4.236	113.6
8	28	1.5	4.236	104.9

(there are no carbonation, drying and wetting cycle, freezing and thawing cycle, and attack of chloride ions involved). Nevertheless, the macro-cracks and micro-cracks were still observed (shown in Figs. 5 and 6), and this result confirms again that the action of delayed ettringite is the dominant factor for the degradation of concrete.

Experimental results also indicate that the degradation of concrete under sulfate attack dose not occurs in the initial stage of sulfate attack process. Contrarily, it was found that in this stage the modulus of concrete increases (shown in Fig. 3). This is because the continuous hydration, which leads to increase of C-S-H gel and repairs the original defects in concrete, and enhance the modulus and strength of the material. Otherwise, the filling effect of DEF can reduce the porosity of concrete, which leads to the increase of modulus.

In the new model for corrosion damage evolution, γ is a nondimensional loading coefficient defined by Eq. (25), and m is a non-dimensional parameter. They are all constant if the material is determined, and this is because the effect of the concentration of sulfate solution on the damage evolution has been denoted by t_{cr} and t_{p} . Other parameter, i.e., the characteristic time, t_{p} , however, changes with the concentration of sulfate solution. This indicates that the rate of chemical reaction strongly affects the damage evolution. Eq. (21) shows that t_n depends on the coefficient of reaction, k. The faster the reaction, the shorter the characteristic time. In order to discuss the effect of such a characteristic time, the numerical calculation is performed. For convenience, we take $\gamma = 1.5$, m = 4.236, and the effect of critical nucleation time on the damage evolution is ignored in the calculation. The results are plotted in Fig. 9. It can be seen that the damage evolution decreases with the increase of t_p . The numerical results of the second derivative of damage with respect to time, d^2D/dt^2 , were plotted in Fig. 10. One can see that d^2D/dt^2 approximately equals zero if $t = t_p$, which means that t_p can be referred to the inflection point of damage evolution.

The damage evolution starts from the critical nucleation time, t_{cr} , which depends on the tensile strength of concrete (see Eqs. (13) and (24)). If the tensile strength of concrete is enhanced, then the speed of corrosion damage is slowed down. In terms of this sequitur, we suggest a new method for improving sulfate resistance of concrete; that is to add short fibers into concrete. The tensile strength of concrete is enhanced in this way [32], while the sulfate attack induced damage is effectively reduced.

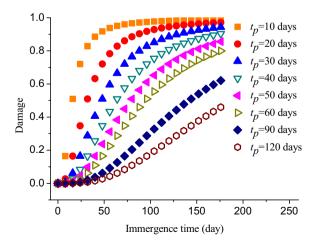


Fig. 9. Effect of t_p on damage evolution.

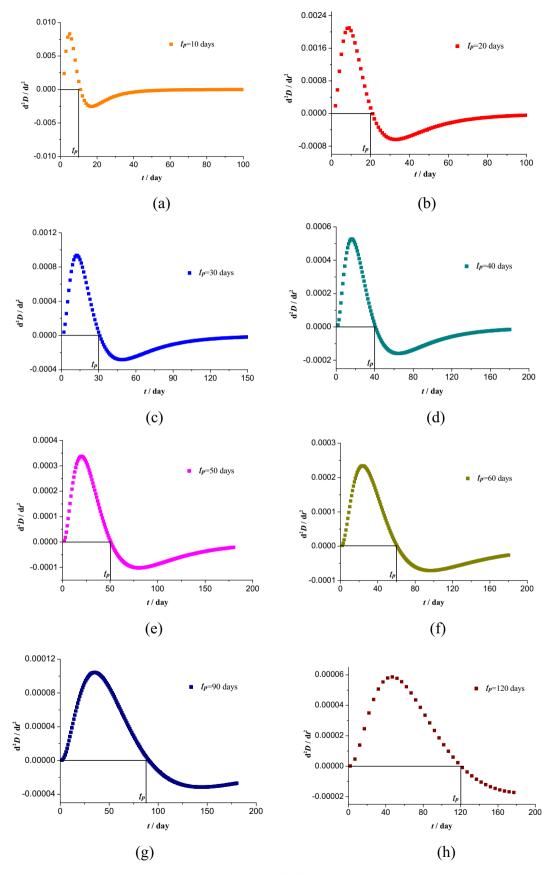


Fig. 10. Plot of $d^2D/dt^2 \sim t$ curves.

5. Conclusions

In this paper, the damage evolution in concrete under sulfate attack is investigated. The experimental results confirm that the expansion stress induced by delayed ettringite is the dominant mechanism for the degradation of concrete. The damage evolution can be divided into two stages, and the elastic modulus of concrete dose not decrease but increases in the first stage. This phenomenon is caused by continuous hydration and the filling effect of delayed ettringite. The mechanism for damage nucleation is analyzed by using Eshelby's equivalent inclusion method. It is found that the tensile stress due to the action of expansive stress produced by delayed ettringite leads to nucleation of micro-cracks. By virtue of this opinion, a new method is proposed, i.e., to increase the tensile strength by adding short fibers for improving the sulfate resistance of concrete. Based on the experimental results, as well as chemical reaction and reaction rate equation, we derive a differential equation for governing the evolution of expansive stress. By assuming that the damage variation obeys Weibull's distribution function, we proposed a new model of damage evolution, which can be used to properly describe the characteristics of corrosion damage of concrete under sulfate attack. The methods used in this study are valid for establishing the strength evolution of concrete attacked by sulfate or other corrosion environment.

Acknowledgments

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