

The Early Hydration Characteristics of Portland Cements with Superplasticizer Using Electrical Measurements

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Abstract In this paper, an electrical measurement was applied to investigate the early hydration characteristics of cement paste with different dosages of superplasticizer. The bulk electrical resistivity development curves and the derivative curves were studied. It is found that there are two main peaks and four critical points on the derivative curves. The critical points are used to divide the hydration process into dissolution, setting, acceleration, deceleration and diffusion-controlled periods. The physical meanings of the critical points are identified by the penetration measurement of Vicat device and the heat evolution test. The setting occurred in the second period which provides an alternative method to determine the setting time by the electrical measurement. The results of the bulk electrical resistivity have a positive relation with that of the compression strength. The retardation effect of the superplasticizer is demonstrated by the delayed time for the critical points occurrence and confirmed by the setting time. A rapid method for selecting a suitable dosage of the chemical admixture for construction requirements is proposed.

Keywords: electrical resistivity, setting time, superplasticizer, hydration

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1. Introduction

Currently, the demand for High Performance Concrete (HPC) is largely increased due to its technical and commercial advantages. Such materials have improved mechanical and durability properties because of the incorporation of chemical and mineral admixtures. Superplasticizer is often used because most concretes are produced with superplasticizers to obtain high workability at a low water cement ratio [1,2]. The excessive retardation of the setting can lead to delay in the removal of formwork and the load application which result in a higher project cost. The optimization and quality control of the admixtures need advanced and accurate monitoring system that can monitor the cement-based material properties in the early stage of hydration. In this research, a non-contacting electrical resistivity monitoring system was used [3]. The advantages of the use of this technique include its accuracy, a high reproducibility, a continuous data acquisition and easy test procedures.

The electrical properties of cement pastes have proved to be sensitive to microstructure changes of cementitious materials [4,5,6]. Electrical resistivity measurements (ERM) has been applied to determine the hydration process and the influence of different admixtures [7,8,9], to determine the aggregate content in hardened concrete by Xie et al [10], and to estimate the strength and ionic

diffusivity [11,12]. The advanced work included predicting hydration properties and conductivity by computer modeling [13].

The objectives of the current research are mainly to analyze the characteristics of the electrical resistivity curve, and to compare the influence of different superplasticizer dosages on the hydration of cement at the same water cement ratio (w/c) by using electrical resistivity measurement.

The unique and significance of this work are as follows. The physical and chemical meanings of the critical points are described and the hydration periods are analyzed with the combination of other methods. The empirical equation to evaluate the setting time is the first proposed based on the duration time of the resistivity critical points. The rapid method to choose the suitable dosage is first proposed according to the construction requirements such as workability and setting time.

2. Materials

Portland cement meeting the requirements of ASTM Type I was used for all samples. The superplasticizer was solid superplasticizer (sp) and β -naphthalene type.

The paste samples were prepared with the superplasticizer/cement (sp/c) dosages of 0 %, 0.5 %, 0.8 % (by solid weight) at a constant water cement ratio (w/c) of 0.3. The sample mixtures are shown in Table 1.

P0.3SP0 stands for water/cement ratio = 0.3 and superplasticizer/cement ratio = 0 %. P0.3SP5 stands for water/cement ratio = 0.3 and superplasticizer/cement ratio

= 0.5%. P0.3SP8 stands for water/cement ratio = 0.3 and superplasticizer/cement ratio = 0.8%. The tests were conducted at 20°C.

Table 1. Mix proportions, setting times and compressive strengths

Code	w/c	Cement	Sp (%)	Setting time (hours)		Compressive strength (MPa)	
				Initial	Final	one day	2 days
P0.3SP0	0.3	1	0.0	3.31	3.98	49.5	61.1
P0.3SP5	0.3	1	0.5	5.50	6.08	45.2	56.1
P0.3SP8	0.3	1	0.8	6.93	7.73	42.9	51.9

3. Methods

3.1. Electrical Resistivity Measurement

The electrical resistivity was measured by a non-contacting electrical resistivity apparatus that was invented by Li and Li, 2003, and manufactured by Hong Kong B.C Technologies. The schematic arrangement is shown in Figure 1, which consists of a generator, amplifier, transformer core, current sensor, data acquisition interface and computer. The generator produces a sine wave with 1 kHz frequency. The amplifier magnifies the sine wave to match the transformer primary coil. The data acquisition interface samples the data which is transferred to the computer. This system can continuously run. The transformer principle is adopted in this apparatus, when voltage is applied to the primary coil, a toroidal voltage V will be generated in the ring sample that acts as the secondary coil of the transformer [3]. The toroidal current, I , can be measured by a leakage current meter and hence the resistivity of the concrete can be calculated through V and I , based on Ohm's law. There are no electrodes in the apparatus so the interface cracks between the electrodes and matrix are eliminated. The pastes were mixed for 2 minutes in a planetary-type mixer at 45 rpm and a further 2 minutes at 90 rpm. After mixing, the mixture was cast into the ring-shaped mould that was kept at 100 % relative humidity during the test period. Following casting, the data were automatically recorded showing the development of electrical resistivity. The sampling interval was one minute, and the recorder was stopped at or after 48 hours.



Figure 1. Picture of the main part of a non-electrode resistivity measurement

3.2. Setting Time

Vicat apparatus was used to test the setting time for w/c=0.3 pastes with different dosages of superplasticizer. Vicat needle penetration tests were conducted. The needle diameter was 1.13 mm, and the probe weight was 300g. The test ring and the glass bottom plate were glued to avoid the leakage of paste. The initial setting time was determined when Vicat needle penetrated 25 mm. The final setting time was the time when the needle did not sink visibly into the paste. The testing temperature was 20°C, and the relative humidity was 95±5 %.

3.3. Heat Evolution

The heat evolutions from all the cement paste mixtures using 10 g cement and appropriate amounts of mixing water were measured in a JAF isothermal conduction calorimeter manufactured by Wexham. Development at 20°C. They were hand-mixed in polythene bags outside the calorimeter for 3 minutes. The heat evolution process was recorded up to 48 hours.

3.4. Compressive strength

Each mixture was cast into 10 cm x 10 cm x 10 cm cubic moulds for compressive strength test. The samples were demolded at 24 hours after casting, and were then transported to the moist room at 20±2°C and 95±5 % relative humidity for curing. The 1 day, and 2 days compressive tests were conducted with a loading rate of 15 MPa per minute in a compression test machine.

3.5. Fluidity of Cement Paste

The Marsh cone test [14] was used to determine the relative fluidity of the cement paste flowing through the spout with a diameter 8 mm. This method is similar to the flow cone tests in the ASTM C 939 for grouts. By measuring the time taken for a certain volume of the paste to flow through the cone. The relative fluidity can be obtained. After mixing the paste for 4 minutes and waiting 2 minutes, 800 ml mixing slurry is poured into the cone, and the time was recorded for first 200 ml of the paste to flow through the cone. The shorter the flow time means the higher fluidity.

4. Results and Discussion

4.1. Four Critical Points and Five Periods on $\rho(t)$ - t and $dp(t)/dt$ - t Curves

The bulk electrical resistivity development with time ($\rho(t)$ - t) for different mixtures is plotted in the lower

part of Figure 2, which corresponds to the secondary y-axis and shows the sigmoid change of electrical resistivity with time up to 48 hours. The derivative curves $\left(\frac{d\rho(t)}{dt} - t\right)$ are displayed in the upper part of Figure 2,

which corresponds to the primary y-axis and provides the rate of resistivity development.

On the derivative curve of sample P0.4SP8, four critical points, P₁, P₂, P₃ and P₄, are labeled. The critical points and physical meanings are summarized as followings:

- (1) Point P₁ (ρ_1, t_1): $\frac{d\rho}{dt} = 0$, corresponding to the first peak point on the derivative curve of the electrical resistivity. It relates to the beginning of the induction period.
- (2) Point P₂ (ρ_2, t_2): the second peak on the derivative curve. It relates with the end of the setting period when the fluid paste starts to stiffen and to gain strength. It the phase transformation from AFt to AFm [15] and is correlated to the initial setting time (t_{ini}) and final setting time (t_{fin}) by the penetration measurement.
- (3) Point P₃ (ρ_3, t_3): the third peak on the derivative curve. It is the transitional point that the kinetics

of reaction is converted to the chemical and diffusion control process. It is close to the peak time (t_q) on the heat evolution.

- (4) Point P₄ (ρ_4, t_4): the inflexion point on the derivative curve. It indicates the start of the diffusion control process. It is confirmed by the result of the heat evolution.

Five periods are readily seen from Figure 2 based on the four critical points. The electrical properties of five periods are summarized in Table 2 and described below.

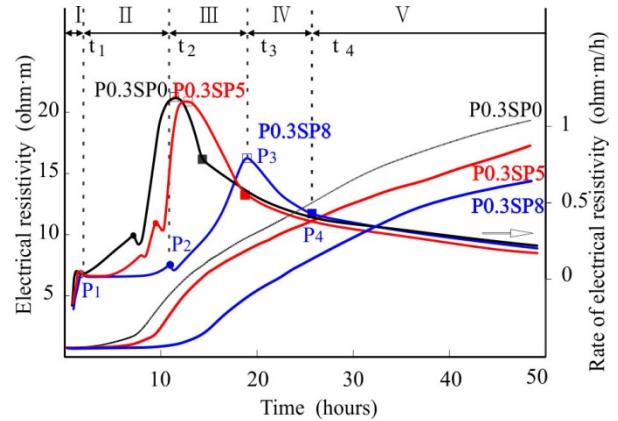


Figure 2. The electrical resistivity curves and derivative curves

Table 2. Characteristics of five periods of hydration process

Period name and characteristics	$\rho(t)-t$	$d\rho(t)/dt-t$	$\rho_0(t)-t$ and $\varphi(t)$	Chemical process and kinetics	Mechanical property	Relevant tests
Dissolution-crystallization period (I)	Decrease	$t \leq t_1$	$\rho_0(t)$ changes dominant, $\varphi(t)$ changes slightly	dissolution of cemented-based materials, chemical control		Liquid solution resistivity t_i
Induction-setting period(II)	Slight increase	$t_1 < t \leq t_2$		coating formation and retarding dissolution, nucleation or diffusion control	initial and final setting	Vicat test t_{ini}, t_{fin}
Acceleration period (III)	Fast increase	$t_2 < t \leq t_3$		accelerating growth of hydrates, chemical control	rate of initial hardening	Heat evolution time, t_q
Deceleration period (IV)	Slow increase	$t_3 < t \leq t_4$		slow and steady formation of hydrates, chemical and diffusion control	rate of early strength gain	Heat evolution time, t_q
Steady period (IV)	Slower increase	$t > t_4$		$0 < d\rho/dt \leq d\rho(i)/dt$, slow formation of hydrates, diffusion control	rate of later strength gain	Heat evolution

- (1) Dissolution-crystallization period (I) is from the mixing time to the first peak point time (t_1). In this period, $\frac{d\rho}{dt} \leq 0$ and the dissolution of cement is dominant. After water is added to the cement, the dissolution of ions from the cement takes place giving out various mobile ions, including potassium (K^+), sodium (Na^+), calcium (Ca^{2+}), hydroxyl ions (OH^-), sulfate (SO_4^{2-}), silicate ions, aluminate ions. These free mobile ions favor the conductivity of the matrix solution at this period.
- (2) Induction-setting period (II) is from the first peak point time (t_1) to the second peak point time (t_2). With the end of the dissolution period, the solution in the paste reaches a supersaturated state and Ettringite, CH and C-S-H are formed on the surface of the cement particles as coating, which delays the hydration [16], and the induction period of hydration commences. With the increase of the hydrates, the coating ruptures due to osmotic pressure [16] and the hydration accelerates and the porosity decreases until the

paste can resistance to the penetration of Vicat needle.

- (3) Acceleration period (III) is from the second peak point time (t_2) to the third peak point time (t_3). In this period, $\frac{d\rho}{dt} > 0$ and the curve displays a rapid increase.
- (4) Deceleration period (IV) is from the third peak point time (t_3) to the inflexion time (t_4). In this period, $\frac{d\rho}{dt} > 0$ and the curve displays a rapid decrease.
- (5) Diffusion-controlled period (IV) begins from the inflexion time (t_4). In this period, $\frac{d\rho}{dt} > 0$ and the curve undergoes a slow decrease and the reaction is diffusion-controlled.

By comparing the curves of samples P0.3SP0, P0.3SP5 and P0.3SP8 in Figure 2, the effect of the superplasticizer on the rate of the electrical resistivity $\left(\frac{d\rho(t)}{dt} - t\right)$ and the

bulk electrical resistivity ($\rho(t)-t$) can be found. The $\frac{d\rho}{dt}$ curves with superplasticizer present their four critical points (P_1, P_2, P_3 and P_4) to shift a longer time in the same period than that of sample P0.3SP0 without superplasticizer. The ρ curves with superplasticizer yield a lower slope rate in the same period with the control sample P0.3SP0. The time in each period (t_1, t_2, t_3, t_4) of sample P0.3SP8 was longer than that of the other two samples. The minimum electrical resistivity varied approximately between $0.73\Omega\text{-m}$ to $0.64\Omega\text{-m}$ for the samples that are close to the results obtained by Tamas et al [15]. The main chemical component of the superplasticizer is naphthalene. The adsorption of the naphthalene on the cement particles creates a diffuse layer that acts as an osmotic membrane and restricts the contact between cement and water, as proposed by Banfill, et al. [16] and Andersen, et al. [17,18,19]. Therefore, the rate of the hydration of cement-based materials decreases with the content of superplasticizer. The relation between the superplasticizer dosage (d) and the time corresponding to the first peak point (t_1) is shown in Equation 1.

$$t_1 = 54.6d + 0.38, R^2 = 0.9576. \quad (1)$$

The retardation is similar to the reference result [20] that the maximum temperature and the time to reach the maximum was linearly depended on the amounts of superplasticizer.

4.2. Setting Time Occurred between the Minimum Point and the First Peak Point

Setting times (t_{ini}, t_{fin}) of the samples by Vicat test are tabulated in Table 1 and shown in Figure 3. There is an increase in the second period on the bulk electrical resistivity curve. This increase is due to the transition process from some open pores to closed ones and the porosity decrease with hydration. The ions in the pore solution become difficult to migrate that result in the increase of the bulk resistivity and setting occurrence.

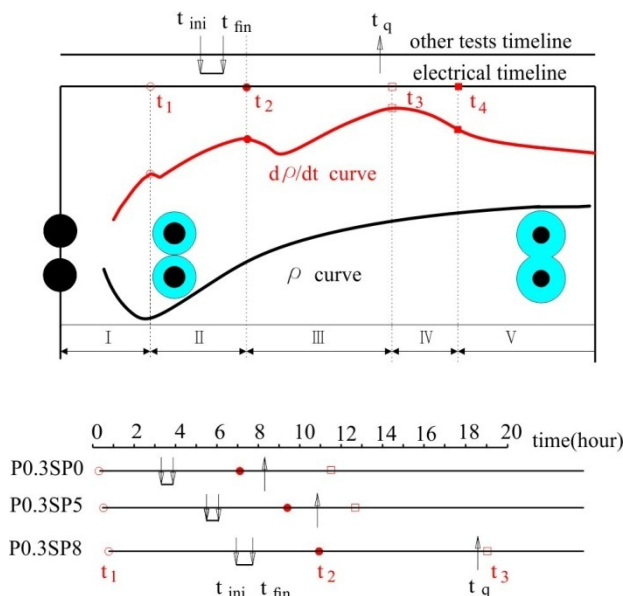


Figure 3. The correlation between the critical point, the setting time and the peak time of the heat rate

It can be easily seen that the setting time occurred in the second period (II) for all samples. By comparing the initial setting time (t_{ini}) and final setting time (t_{fin}) with the time duration of the second period (Δt_{12}) shown in Figure 3, the correlation can be found between the duration time Δt_{12} from electrical measurement and the setting time from Vicat as shown in Equation 2.

$$\text{Setting time}(t_{ini}, t_{fin}) \approx t_m + (1/2 \sim 2/3)\Delta t_{12}. \quad (2)$$

The result also shows that samples P0.3SP5 and P0.3SP8 with superplasticizer always have the longer both setting time and the electrical critical point time as compared to the control sample P0.3SP0.

It is demonstrated that the superplasticizer dosages have an effect in prolonging the setting time and hydration process that reflects the retardation of hydration with superplasticizer. The overdose of superplasticizer causes an excessive retardation of the setting that results in higher project cost. The electrical resistivity provides a rapid method to select a suitable dosage of admixture.

After finding the correlation of the duration time Δt_{12} from the electrical measurement and the setting time, the relation can be used to choose the suitable dosages of admixtures according to the construction requirements. When Δt_{12} is shorter, the cement-based material has a higher reaction rate and it shortens the demolding time and accelerates the progress of construction. On the contrary, when Δt_{12} is longer, the cement-based material has a lower reaction rate and it can prevent the construction from cracking due to the heat from the large volume cement-based material.

4.3. Heat and Correlation with the Electrical Resistivity

The hydration process of cement is a transformation from a high energy state to a lower state, and this process is evolved with heat release. Evolution of the hydration heat can therefore reveal information about the chemical reactions taking place in the cement hydration. Conduction calorimetry is a traditional and useful method to study the hydration behavior of different effects on hydration, such as by superplasticizers.

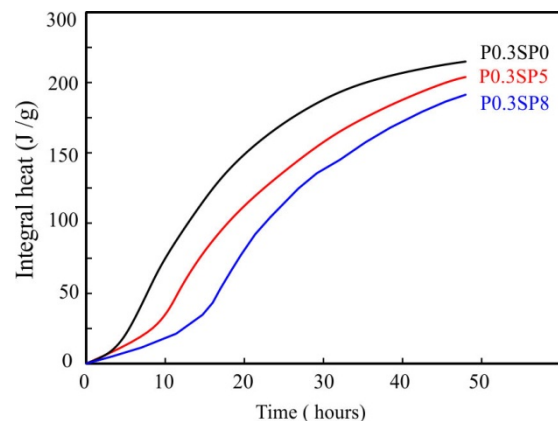


Figure 4. Heat evolution of P0.3SP0, P0.3SP5, P0.3SP8 during 48 hours

Figure 4 shows the curve of the integral amount of heat developed up to 48 hours. Figure 5 gives the curve of the rate of heat developed to 48 hours. Figure 6 shows the

different the times, which are t_2 and t_3 in Figure 2 from the electrical measurement, t_q to reach the maximum heat rate in Figure 5 and setting time (t_{ini} , t_{fin}) from Vicat test, respectively versus the contents of superplasticizer. From Figure 4, the total heat of the samples P0.3SP5 and P0.3SP8 with superplasticizer after 48 hours is a little lower than that of the control sample P0.3SP0. $\rho(t)$, at which the 48 hours shows the same order.

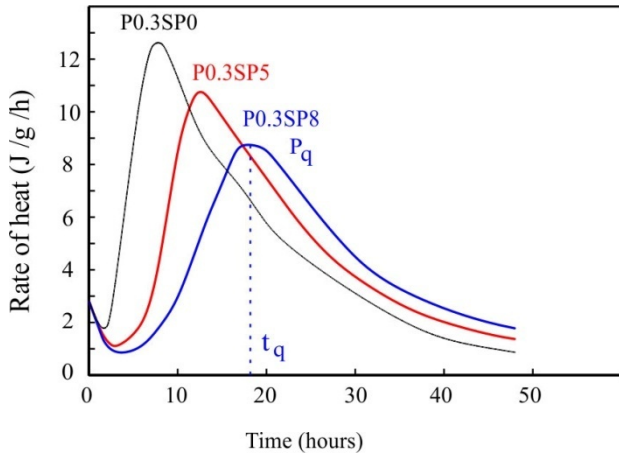


Figure 5. Rate of heat evolution of P0.3SP0, P0.3SP5, P0.3SP8 during 48 hours

From Figure 5, the time (t_q) to attain the maximum heat has been increased from 8.1 hours for the control sample P0.3SP0 to 12.8 hours for sample P0.3SP5 and 18.6 hours for sample P0.3SP8. It can be seen that the maximum rate of heat evolution increases with an increase in superplasticizer dosage, and the time to reach the maximum rate is longer for the higher dosage. With the increase of the superplasticizer, the hydration degree is decreased at early age for the same curing time.

The t_q values for the different samples are compared with the timeline of the electrical resistivity critical points in Figure 3. It is noted that t_q is located in the induction-setting period III as in Figures 3 and 6. It indicates the good correlation between the electrical measurement and heat evolution measurement. Peak point P_i on the resistivity curve has the similar kinetics meaning to the peak point P_q on the heat evolution curve. P_i represents the transformation from a chemical reaction to a chemical-diffusion control period.

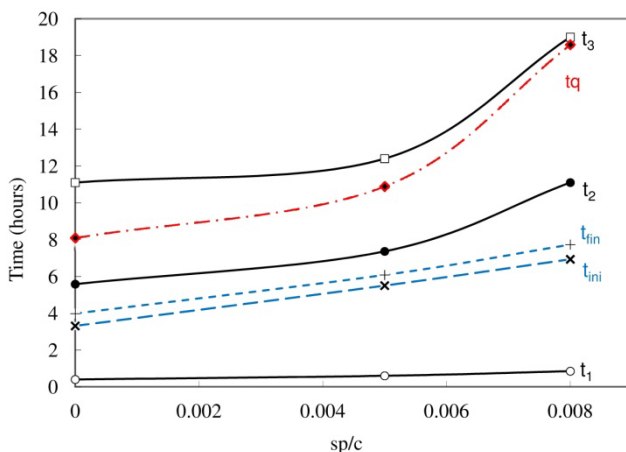


Figure 6. The times to reach the critical points, setting time vs. the content of superplasticizer

4.4. Fluidity

Superplasticizers is incorporated in cement-based materials in order to reduce the water cement ratio for strength increase with maintaining workability, or to increase the workability at a constant water cement ratio [21]. In this research, the superplasticizer was used to increase workability. The fluidity increases with the increase of superplasticizer amounts confirmed by the results of the Marsh Cone test.

Sample P0.3SP0 cannot flow out from the Marsh cone due to the lower fluidity. The Marsh cone flow times for samples P0.3SP5 and P0.3SP8 are 17 seconds, and 8 seconds, respectively. It is reasonable that the fluidity is increased with the increase of superplasticizer for the same water/cement ratio. The increased workability is relative with the easy cement particles movement due to the organic chemical molecules in the superplasticizer break the flocculated cement and the trapped water releasing.

4.5. Compressive Strength

Compressive strength data, tabulated in Table 1, is one of the most important mechanical properties of cement-based materials. Pastes P0.3SP5 and P0.3SP8 with superplasticizer have lower compressive strengths than the control sample P0.3SP0 at 1 day and 2 days. The higher the dosage of superplasticizer, the lower the compressive strength and the lower resistivity at 1 day and 2 days are obtained for the same water / cement ratio system. Thus, there is a positive relation between the compression strength and resistivity.

Properties of porous cement-based material, such as mechanical strength and durability, depend primarily on the pore space and the morphology of the solid phase. The development of electrical resistivity with time reflects the decrease of pore space with conductive ions and the increase of solid space with insulator properties for the same initial porosity with a similar system.

In summary, the critical points on the electrical resistivity timeline are strongly relative with the setting time, the time of the resistivity critical point and the heat rate evolution peak time. The electrical resistivity measurement can not only provide information on the period of setting and hardening occurrence, but also give more details and more accurate information on the overall period of hydration. The electrical resistivity with time reflects the kinetic of microstructure of cement-based materials.

5. Conclusions

1. The bulk electrical resistivity development curve ($\rho(t)-t$) is a type of s-shaped growth curve. The curve begins with a slow start, followed by a rapid increase, then a slow rise. The rapid increase corresponds to the setting and hardening of the cement-based material.
2. There are three main peaks on the bulk resistivity differential development curve $\left(\frac{d\rho(t)}{dt}-t\right)$. The

second peak has a rheology physical meaning, i.e. the loss of the fluidity. The third peak has a chemical kinetic meaning that is the transformation from the chemical control to the chemical-diffusion control reaction. The physical meanings of the critical points are confirmed by the penetration measurement of Vicat test and the heat evolution.

3. The hydration process can be divided into a dissolution-crystallization period, an induction-setting period, an acceleration period, a deceleration period and a steady period. An evaluation concerning the early strength gains can be alternatively replaced by the electrical resistivity measurement.
4. The setting times occurred before the second peak time. It gives an alternative method to determine the setting time based on the derivative curve of the electrical resistivity.
5. The superplasticizer prolongs all the periods of hydration for the same water / cement ratio paste. The bulk electrical resistivity measurement provides a rapid method for selection suitable dosage of a chemical admixture for construction requirements.

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