

Research Article

Effect of Particle Size of Periclase on the Periclase Hydration and Expansion of Low-Heat Portland Cement Pastes

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In this paper, low-heat Portland cement (LHC) clinkers were prepared by calcining raw materials at 1350°C for 2.0 hours, 1400°C for 1.0 hour, 1400°C for 1.5 hours, 1400°C for 2.0 hours, 1450°C for 1.0 hour, and 1450°C for 2.0 hours. The clinkers were ground with gypsum to produce LHC. The particle size of periclase was analysed by BSEM. Expansion of LHC pastes due to hydration of periclase was measured. The hydration degree of periclase in LHC pastes was quantitatively determined by XRD internal standard method and BSEM. The results showed that the particle size of periclase was larger when clinkers were calcined at higher temperatures or for longer time. Smaller periclase (2.60 μm) in LHC pastes tended to hydrate faster. As a result, expansion of LHC pastes develops relatively faster. Smaller particle of periclase in clinker tends to result in higher hydration degree of periclase in pastes cured at 20°C for 240 days, and there is a small amount of brucite appearing around periclase. The hydration rate of 4.00 μm periclase particle in cement paste cured at 80°C is obviously faster than that in paste cured at 20°C and 40°C. When cement paste was cured at 80°C for 7 days, the periclase was hydrated for 32.56%. The smaller size periclase (1–3 μm) had fully hydrated when the curing age was 240 days, and a large amount of brucite was produced around the larger periclase particle.

1. Introduction

Low-heat Portland cement (LHC), namely, high belite cement [1], is currently attracting a great deal of interest worldwide. This is largely due to its lower energy consumption, CO₂ emission, and heat release than ordinary Portland cements (OPC) and moderate-heat Portland cements (MHC) [2]. LHC has also many advantages, such as higher long-term strength and smaller dry shrinkage [3, 4]. The MHC is not only one of the largest amount of special cement but also the main binder used in hydraulic concrete in China, amounting for approximately 30% of hydraulic engineering cement so far [5]. Yang et al. [4] indicated that LHC concrete has a better anticrack behavior than MHC concrete. In recent years, LHC has been used in several large hydraulic structures, for example, the Three Gorges Dam, because of its low hydration heat and excellent durability [6, 7].

Like conventional Portland cement, MgO as a minor component exists in LHC. MgO as an expansive additive is

often used in dam construction to compensate for the slight natural shrinkage of PC during hydration, which can continue for months or years in service [8, 9]. As we all know, the free MgO in the form of the periclase crystal in clinker convert to Mg(OH)₂ with water, and the solid volume expansion increases 118% [10]. However, the presence of excessive amounts of MgO in the hydraulic concrete will lead to the volume expansion [4] and cause the crack formation of the dam. It is an effective and economic method to prevent the crack of dam by using the delayed expansion of periclase to compensate the thermal shrinkage of the concrete [11].

Chen et al. [12] and Lou et al. [13] found the characteristics of delayed expansion of the periclase, which indicated the expansion occurred mainly in the later period. The results of Mo et al. [14] showed that higher calcination temperature and longer residence time will cause grain growth of MgO, thus decrease in inner pore volume and specific surface area, thereby decreasing the hydration

activity of MEA. Mehta et al. [15] thought that the expansion rate of the MgO in cement can be artificially adjusted by controlling the calcination temperature and particle size of the periclase. Song et al. [16] reported that periclase content increased with the increase of MgO content, periclase hydrated at a higher rate at early age, and the increase of curing temperature accelerated the hydration rate. There were results showing that increasing the calcination temperature and prolonging holding time had little effect on the whole content of f-MgO in high magnesium clinker, but the number of the larger size particles of the periclase crystal had increased [17, 18].

Periclase in Portland cement clinkers may cause unsound issues to concrete, and its content is often limited in many specifications of cements [19–21]. The expansion due to hydration of periclase in MHC clinkers [22] and especially in Mg-based expansive agents has been successfully used to compensate shrinkage due to decrease of temperature in dam concretes [23–25]. Then, there are many researchers to investigate the expansion control of MgO expansive agent [14, 26]. However, the expansion control of periclase in LHC has not been deeply studied.

In order to achieve the compensation effect on the temperature shrinkage of hydraulic concrete by using the delayed expansion of periclase in the LHC cement, LHC clinker was prepared under different calcination conditions, and the influence of crystal size of periclase on the hydration and expansion of the self-prepared low-heat cement was studied in this paper.

2. Materials and Methods

2.1. Materials. The low-heat Portland cement (LHC) clinkers used were prepared in the laboratory by calcining raw materials of limestone, silica, dolomite, aluminum ore, and copper slag that came from Sichuan Jiahua Special Cement Company. The gypsum used was from Huaneng Power Plant in Nanjing. Table 1 shows the chemical compositions of raw materials. XRD analysis of gypsum is shown in Figure 1. The raw materials were, respectively, calcined at 1350°C for 2.0 hours, 1400°C for 1.0 hour, 1400°C for 1.5 hours, 1400°C for 2.0 hours, 1450°C for 1.0 hour, and 1450°C for 2.0 hours. The theoretic mineral compositions of LHC clinker are shown in Table 2.

The obtained LHC clinkers were ground into powders with less than 10% sieve residue. 95% clinker powders and 5% gypsum were put into the mixing bucket, and the mixture was blended with 12 hours to obtain LHC.

2.2. Test Methods

2.2.1. Expansion Test of Cement Pastes. The cement pastes were prepared according to the JC/T 313-2009 (Chinese Standard). The fresh pastes were cast into a mould of 20 mm × 20 mm × 80 mm. The w/c ratio was set at 0.27. The hydration samples were made by casting the pastes into the mould of 20 mm × 20 mm × 20 mm while molding expansion specimens. The pastes with the mould were cured in a moist environment (98% relative humidity) at 20 ± 1°C for

24 ± 2 h, and then demoulded to measure the initial length L_0 . The demoulded specimens were cured in water at 20°C, 30°C, 40°C, and 80°C, respectively. The length L_t of pastes were measured at specific intervals. The expansion of the cement pastes was calculated according to the formula given in Chinese Standard (JC/T 313-2009).

2.2.2. Determination of the Content of Periclase in LHC Pastes. The XRD (Smart Lab, Rigaku, Tokyo, Japan) internal standard method [27] was employed to determine the content of periclase in LHC clinkers and the hydrated paste samples. The internal standard substance and target component were ZnO (AR, $2\theta = 36.50^\circ$) and periclase ($2\theta = 42.90^\circ$). The pure phase periclase crystal was obtained by calcining magnesium carbonate basic at 1000°C for 1.0 hour. The content of ZnO was 1%, and the content of periclase was 1%, 2%, 4%, 6%, 8%, and 10%, respectively. Step scanning was conducted from 35° to 45°, using a step size of 0.02° and a scan speed of 1°/min. The XRD results were fitted by Jade6.0 to obtain the characteristic peak intensity. Using $I_{\text{MgO}}/I_{\text{ZnO}}$ as abscissa axis and MgO percentage as ordinate axis to draw standard working curve, the equation is as follows:

$$Y = 0.49927 + 1.60929X \quad (R^2 = 0.9968). \quad (1)$$

The oven-dry paste samples were ground into a powder through an 80 μm sieve and then well mixed with 1% ZnO. The hydrated paste was calcined at 950°C for 3.0 hours to remove the bound water to achieve the normalization of the quantification data.

2.2.3. Determination of Particle Size of Periclase in Clinkers and Analysis of Periclase in LHC Pastes. The median sizes of periclase in the LHC clinkers were got by the statistical methods based on the images of FE-SEM (Nava NanoSEM 450, FEI, Oregon State, USA). The FE-SEM was also used to analyse the hydration process of the periclase in cement paste. For BSEM image analysis, the dried slice samples of the cement clinker and the hydrated cement pastes were impregnated with epoxy resin in order to block in the hole. After the epoxy hardened, the samples were polished with 240, 400, 600, 2400, and 4000 grit abrasive paper and then polished using polishing liquid to the mirror surface by Automatic Grinder Polisher (EcoMet 250, Buehler, Illinois, USA).

3. Results and Discussion

3.1. Particle Size and Content of Periclase in LHC Clinkers. Figure 2 shows the BSEM images of LHC clinkers calcined at 1350°C for 2 hours, 1400°C for 1.0 hour, 1400°C for 1.5 hours, 1400°C for 2.0 hours, 1450°C for 1.0 hour, and 1450°C for 2.0 hours. According to the EDS analysis, the deep color particle is periclase in Figure 2. The shape of periclase in LHC clinkers was mostly polygonal or circular with clear boundaries, and it did not vary with calcination conditions changing. The median sizes and contents of periclase in LHC clinkers are shown in Table 3. The average particle size of

TABLE 1: Chemical compositions of raw materials (by weight, %).

Materials	LOI*	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	Ratio
Limestone	41.76	4.03	0.72	0.50	50.29	1.65	0.10	0.02	0.04	67.04
Silica	1.66	87.16	2.41	5.02	1.59	1.52	0.12	0.09	0.07	10.23
Dolomite	43.00	5.04	1.11	0.66	29.60	19.80	0.20	0.08	0.03	15.09
Aluminum ore	14.72	30.48	28.36	21.64	1.86	1.46	0.73	0.24	0.00	5.77
Copper slag	0.00	26.58	6.57	45.80	12.37	5.78	0.24	0.08	0.06	1.87
Gypsum	6.36	2.88	0.32	0.25	33.14	0.33	—	—	42.54	—

*Loss on ignition.

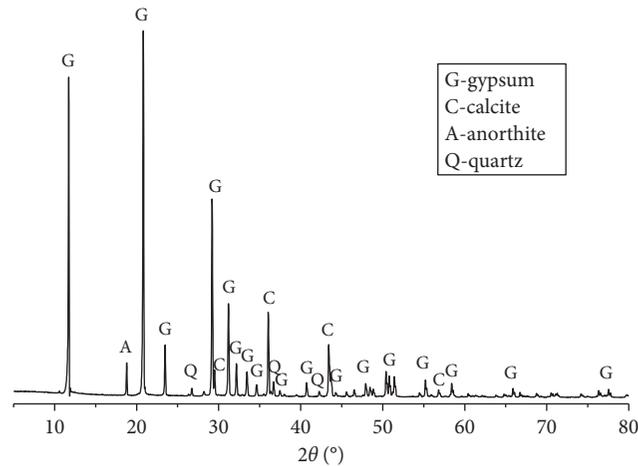


FIGURE 1: X-ray diffraction (XRD) pattern of gypsum.

TABLE 2: Theoretic mineral composition of LHC clinkers (%).

	C ₃ S	C ₂ S	C ₃ A	C ₄ AF	KH	SM	IM
Clinker	34.99	40.01	3.01	15.00	0.80	2.64	0.87

periclase in LHC clinkers calcined at 1400°C and 1450°C for 1.0 hour is 2.60 and 3.92 μm, respectively. The size of periclase in LHC clinkers calcined at 1350°C, 1400°C, and 1450°C for 2.0 hours is 2.94, 3.00, and 4.00 μm. Higher calcination temperature tends to increase the average particle size of periclase in LHC clinkers. The average particle size of periclase in LHC clinkers calcined at 1400°C for 1.0, 1.5, and 2.0 hours is 2.60, 2.80, and 3.00 μm, respectively. Longer holding time will enlarge the size of periclase in LHC clinkers. LHC clinkers calcined at 1350°C for 2.0 hours and at 1450°C for 2.0 hours contain the largest and smallest contents of periclase, being 4.15% and 3.56%. Higher calcination temperature or longer holding time tends to reduce slightly the content of periclase in LHC clinkers.

3.2. Expansion of LHC Pastes. Figure 3 shows expansion of LHC pastes cured in water at 20°C, 30°C, 40°C, and 80°C. It can be seen from Figures 3(a), 3(b), and 3(c), at the same curing temperature, the expansion of cement pastes with 1400°C calcination temperature was the largest when the LHC clinker were calcined for 2.0 hours at 1350°C, 1400°C, and 1450°C, respectively. Both of cement pastes calcined at 1350°C and 1400°C for 2.0 hours and 1.0 hour began to

shrink when they were cured at 20°C for 200 days, and the expansion of cement pastes was -0.0102% and -0.00467%. While the cement clinkers were calcined at 1400°C for 1.0 hour, 1.5 hours, and 2.0 hours, respectively, the expansion of cement paste calcined at 1400°C for 2.0 hours cured at 20°C was bigger than others, the expansion of cement paste calcined at 1400°C for 1.5 hours cured at 30°C was larger than others, and the expansion of cement paste calcined at 1400°C for 1.0 hours cured at 40°C and 80°C was the largest. So it could be concluded that different calcination time periods had little effect on the expansion of LHC pastes. When cement paste calcined at 1400°C for 1 hour was cured in 40°C and 80°C water, the maximum expansion of the cement paste was up to 0.064% and 0.131%. At 20°C, 30°C, and 40°C curing conditions, the expansion of cement paste calcined at 1450°C for 2.0 hours was larger than that of cement paste calcined at 1450°C for 1.0 hour. The expansion of cement paste prepared at 1450°C for 1.5 hours was close to 0% when pastes were cured at 20°C for 240 days. Combining Table 3 and Figure 3, we can conclude that when the calcination temperature of the LHC clinker is lower, the cement paste has greater expansion with the smaller particle size of the periclase, and the LHC has higher content of periclase.

3.3. Hydration of Periclase in LHC Pastes

3.3.1. XRD Internal Standard Method for the Determination of Periclase. Figure 4 listed the XRD patterns of the dried cement pastes of LHC with 2.60, 3.92, and 4.00 μm periclase

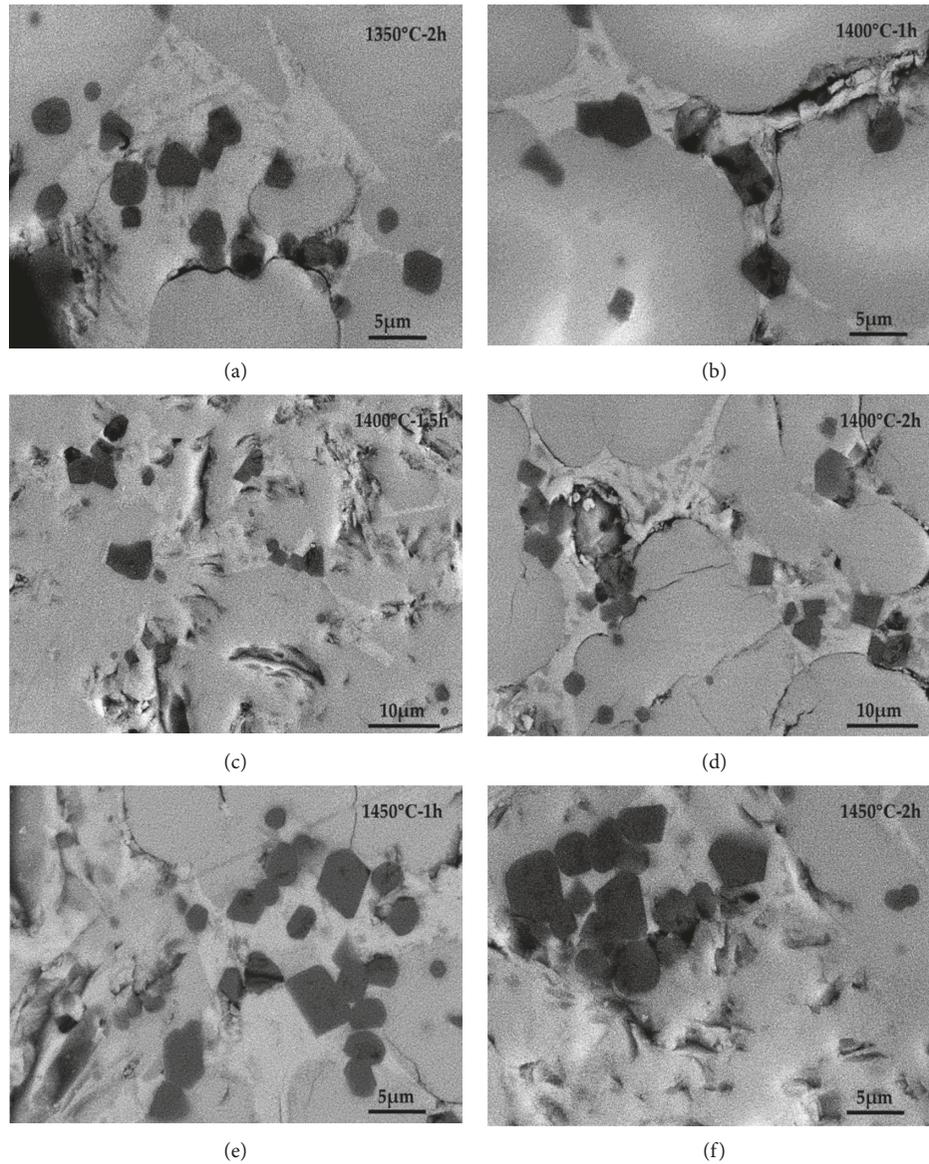


FIGURE 2: BSEM images of LHC clinkers calcined at 1350°C for 2.0 hours (a), 1400°C for 1.0 hour (b), 1400°C for 1.5 hours (c), 1400°C for 2.0 hours (d), 1450°C for 1.0 hour (e), and 1450°C for 2.0 hours (f) (the deep color particles represent periclase).

TABLE 3: The particle size and the content of periclase in LHC clinkers calcined at different conditions.

Calcination conditions	1.0 hour			2.0 hours		1400°C		
	1400°C	1450°C	1350°C	1400°C	1450°C	1.0 hour	1.5 hours	2.0 hours
Particle size of periclase (μm)	2.60	3.92	2.94	3.00	4.00	2.60	2.82	3.00
Content of periclase (wt./%)	3.91	3.72	4.13	3.65	3.56	3.91	3.81	3.65

size which were hydrated at 20°C, 40°C, and 80°C water for 240 days. According to Figure 4(a), it can be seen that there was no obvious diffraction peak of brucite generated until cement pastes were cured at 20°C for 240 days. As Figure 4(b) shows that the very obvious characteristic peak of brucite was observed when the cement paste with 4.00 μm periclase particle was cured at 40°C and 80°C for 240 days, and the characteristic peak intensity of $\text{Mg}(\text{OH})_2$ became higher and higher with the increase of curing temperature.

The XRD internal standard method was used to determine the content of periclase in cement pastes. The hydration degrees of periclase are shown in Table 4. It can be seen from Table 4 that the periclase in cement pastes had not hydrated greatly when the cement pastes were cured at 20°C for 7 days, and the hydration degrees of periclase with particle sizes of 2.60, 3.92, and 4.00 μm were 0.27%, 6.80%, and 6.69%, respectively. Before cured for 90 days, the hydration degree of periclase in cement paste was smaller with

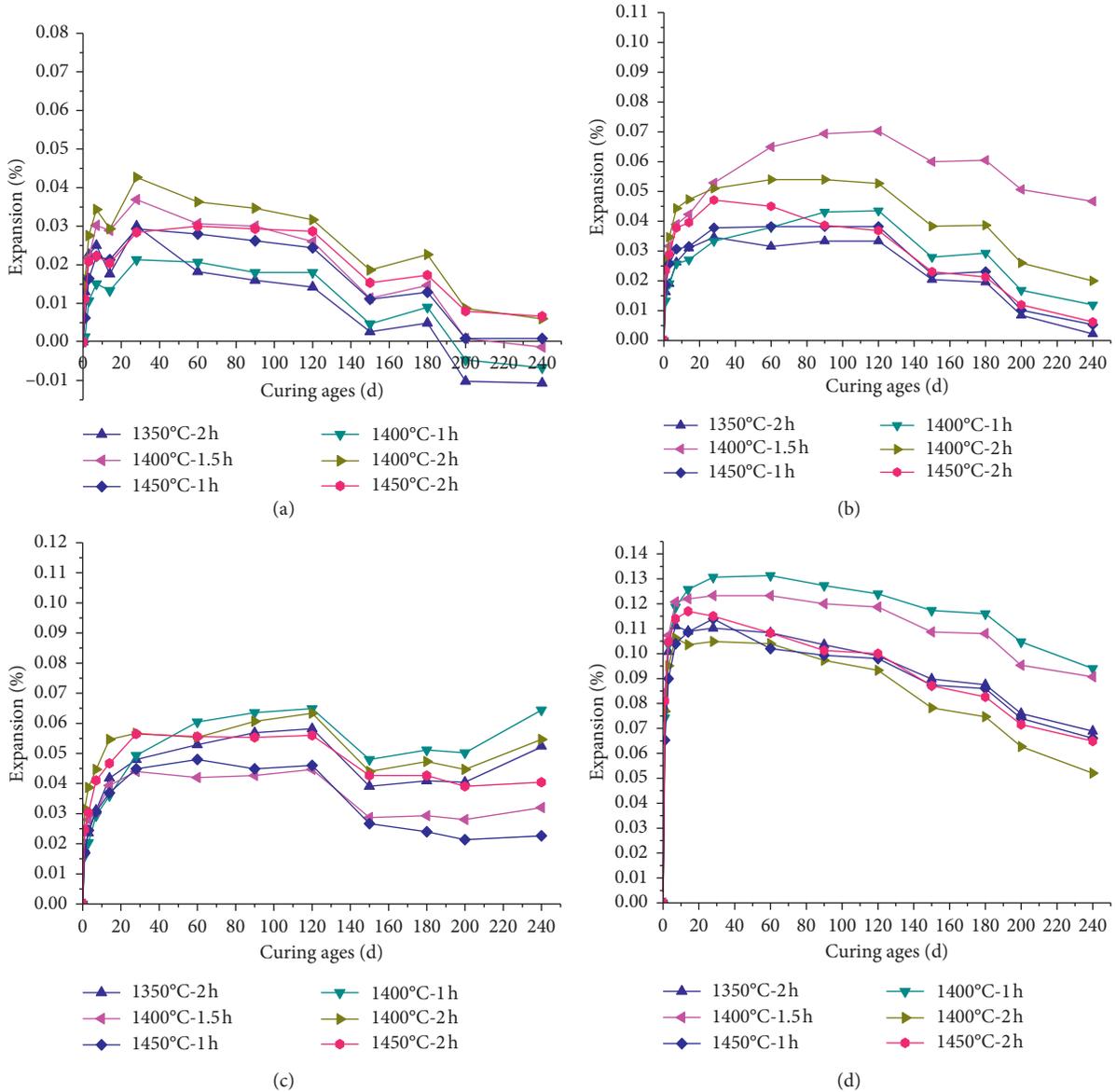


FIGURE 3: The expansion of LHC pastes prepared at different calcination conditions: (a) 20°C, (b) 30°C, (c) 40°C, and (d) 80°C.

the particle size of periclase decreasing. After 90 days, the periclase of 2.60 μm has the highest hydration degree although the periclase size is the smallest in three kinds of cement pastes. At the age of 240 days, the hydration degrees of periclase with particle sizes of 2.60, 3.92, and 4.00 μm were 30.73%, 24.36%, and 28.20%, respectively. When the LHC pastes were cured at 40°C and 80°C for 60 days, the hydration degrees of periclase with 4.00 μm particle size decreased rapidly with the delay of the curing ages, and the hydration degrees were 36.63% and 61.92%, respectively. After 60 days, the hydration degree increased a little with the prolongation of curing ages.

Combining Figure 4 and Table 4, the conclusion can be drawn that the hydration speed of periclase of cement paste cured at 20°C for 240 days was faster with the smaller particle size of the periclase in cement paste obtained at lower calcination temperature. The periclase hydration needed

much more time when cement paste was cured at 20°C because of the slow hydration speed. And the hydration degree of periclase with a particle size of 4.00 μm in cement paste cured at 20°C for 240 days was 28.20% which was much lower than that of periclase cured at 80°C with 67.15%. As mentioned in references [10, 14, 19], the hydration degree of periclase became smaller with the larger particle size of periclase and the increase of curing temperature.

3.3.2. BSEM Images of Cement Paste. BSEM images of cement paste with 4.00 μm periclase particles that cured at 20°C, 40°C, and 80°C for 60, 180, and 240 days, respectively, are shown in Figure 5. P represents the periclase particles, and B indicates the $\text{Mg}(\text{OH})_2$ particles. Figure 5(a) shows that there was no obvious hydration marks around the periclase in the cement paste when it was cured at 20°C for 60

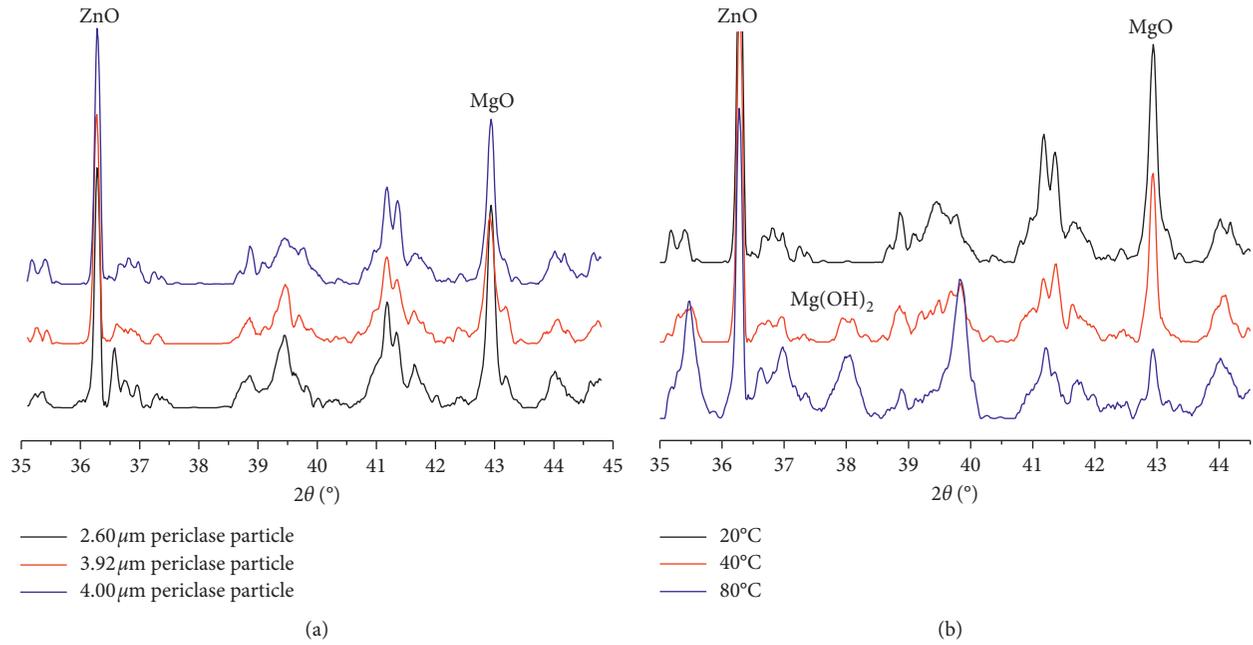


FIGURE 4: The XRD patterns of cement pastes cured for 240 days at 20°C and 80°C, respectively. (a) The cement pastes with 2.60, 3.92, and 4.00 μm periclase particle cured at 20°C for 240 days; (b) the cement paste with 4.00 μm periclase particle cured at 20°C, 40°C, and 80°C for 240 days.

TABLE 4: Hydration degree of periclase in LHC cement pastes cured for 7, 28, 60, 90, 180, and 240 d (%).

Particle size of periclase (μm)	Curing temperature, T (°C)	The hydration degree of periclase (%)						
		0 d	7 d	28 d	60 d	90 d	180 d	240 d
2.60	20	0.00	0.27	3.77	4.85	16.71	29.38	30.73
3.92	20	0.00	6.80	11.05	11.33	15.86	19.55	24.36
4.00	20	0.00	6.69	11.63	12.79	16.28	25.29	28.20
4.00	40	0.00	8.43	25.87	36.63	39.24	45.93	45.93
4.00	80	0.00	32.56	48.55	61.92	63.66	66.57	67.15

days, which indicated that the periclase did not react with water in the early curing ages. In addition, the periclase wrapped in the silicate phase had not hydrated.

From Figure 5(b), it can be seen that when the cement paste was cured at 20°C for 180 days, the color of a few regions around the periclase particles in the backscattered electron image was deepened. Thus, the periclase of cement paste reacted with water to produce a little Mg(OH)₂ that can be judged while the cement paste was cured at 20°C for 180 days. But the color of the surrounding area of periclase particles of the cement paste cured for 240 days was much deeper, and a small amount of brucite was generated at 20°C of water.

There was obvious production of Mg(OH)₂ around periclase in cement paste cured at 40°C for 240 days, which can be seen from Figure 5(d). The periclase in cement paste had hydrated a lot in the 80°C water-curing condition. The small-size periclase had hydrated completely, and a large amount of dense brucite was generated around the large particles of the periclase to form a protective layer around the periclase, which slowed down the hydration speed of periclase.

According to the aforementioned phenomenon, it can be concluded that the periclase hydrated slowly at 20°C. The periclase will hydrate quickly to produce more brucite with the increasing of curing ages and temperature.

4. Conclusions

In order to study the effect of the particle size of periclase on the expansion and hydration of low-heat cement, the expansion of cement paste, hydration degree of periclase, and particle size of periclase were evaluated by testing the length of specimens, XRD internal standard method, and BSEM, respectively. The main conclusions in this paper can be drawn as follows:

- (1) The particle size of periclase was bigger with the holding time increasing at the same calcination temperature. When the residence time was kept consistent, the higher the calcination temperature of the cement linker was, the greater the size of the periclase particle was. And the particle size of periclase in the LHC clinker calcined at 1450°C for 2 h was the largest (4.00 μm).

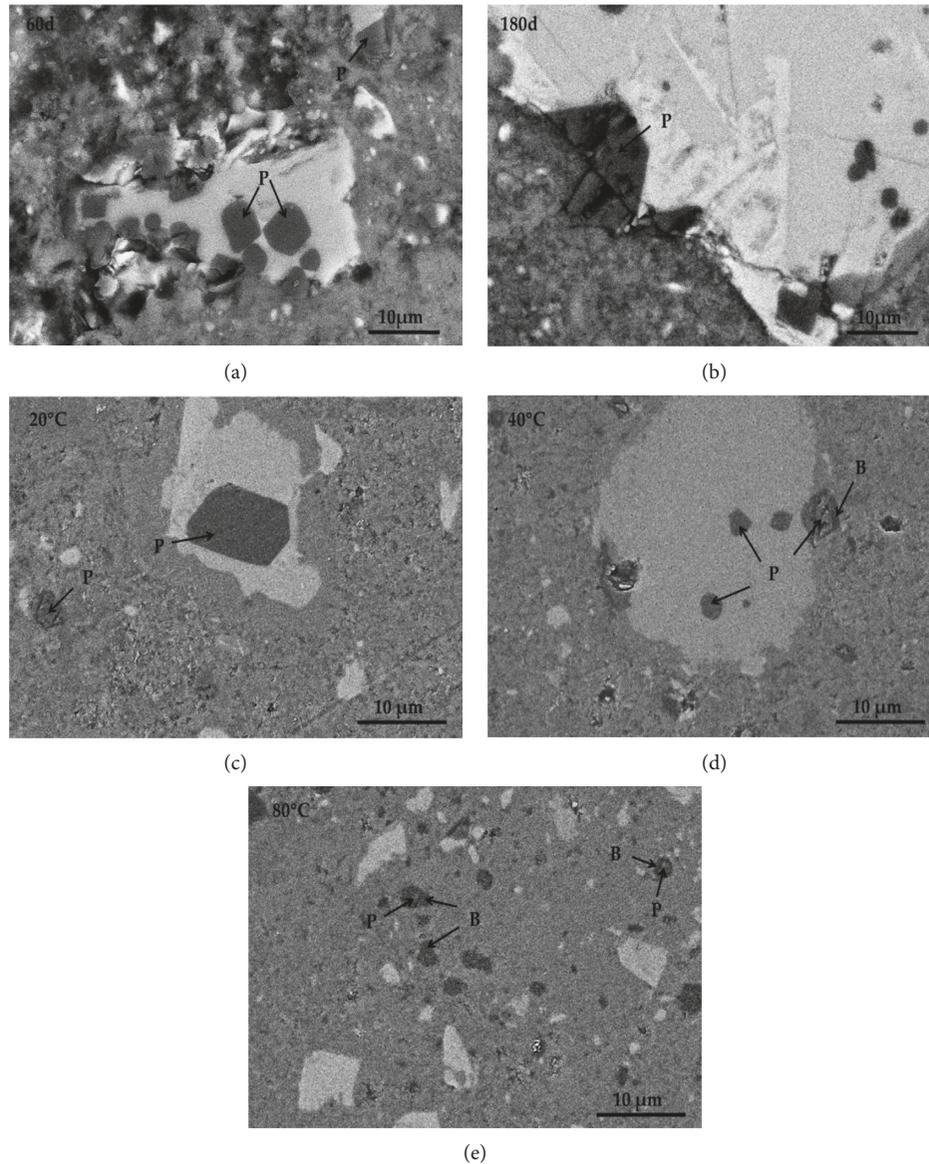


FIGURE 5: BSEM images of cement paste with $4.00\ \mu\text{m}$ periclase particles cured at different temperatures for 60, 180, and 240 days. (a) Curing at 20°C for 60 days; (b) curing at 20°C for 180 days; (c) curing at 20°C for 240 days; (d) curing at 40°C for 240 days; (e) curing at 80°C for 240 days.

- (2) The cement paste has greater expansion than others when the particle size of the periclase in LHC clinker became smaller. The expansion of cement paste with the $4.00\ \mu\text{m}$ particle size of periclase was smaller than the others with the smaller periclase particle size when the cement paste was cured for 240 days at 20°C , 30°C , and 40°C , respectively. For the same curing age, the expansion of the cement pastes cured at 80°C was greater than that of pastes cured under other curing conditions. Shrinkage of the cement paste cured at 20°C water occurred first.
- (3) The hydration speed of periclase in cement paste cured at 20°C for 240 days was higher with the smaller particle size of the periclase. And periclase needed much more time to hydrate when cement paste was cured at 20°C because of the slow hydration

speed. The speed of periclase hydration was faster under 80°C than that at 20°C and 40°C , and the hydration degree of periclase with the $4.00\ \mu\text{m}$ particle size was 32.56% under 80°C cured for 7 days while the periclase had not hydrated at 20°C cured for 7 days.

- (4) The periclase hydrated a lot in the 80°C water-curing condition for 240 days. And the small-size periclase had hydrated completely, and a large amount of dense brucite was generated around the large particles of the periclase when the curing temperature was 80°C .

Data Availability

All data generated or analysed during this study are included in this published article. And readers can access all data used

to support conclusions of the current study from the corresponding author upon request.

Conflicts of Interest

The authors declare no conflicts of interest.

Authors' Contributions

Man Yan and Chen Wang designed and conducted the experimental program. Min Deng provided and designed the project. Zhiyang Chen helped to do experiment and gave many writing suggestions. All authors contributed to the analysis and conclusion.

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