Influence of Curing Temperature on the Deformation properties of Magnesium Oxide Micro-expansive Concrete

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Abstract: The influence of curing temperature on the deformation properties of magnesium oxide(MgO) micro-expansive concrete were studied.Hydration mechanism and expansion characteristics of magnesium oxide(MgO) in cement paste were analyzed by XRD、DSC-TG and SEM. Results indicated.Curing temperature had a great effect on free expansion ratio of MgO mortar, the higher the curing temperature, the bigger the early expansion, the sooner the inflation curves to stabilize.The rising of curing temperature accelerated the hydration rate of MgO and the formation of $Mg(OH)_2$ crystals, because the increased temperature accelerated migration rate of $Mg^{2+}$and OH^-after the MgO hydrating ,and made the formation of $Mg(OH)_2$ crystal to be accelerated accordingly and expansion effect in advance.With the risng of curing temperature, there were some changes in microstructure of $Mg(OH)_2$,which the short lamellar crystals turned into fibrous.

Introduction

Expansion of MgO style micro expansive concrete has delayed expansion without relaxation of expansion stress, and is in line with the characteristics of mass concrete volume changes, so it is the effective measure to hydraulic mass concrete temperature control and crack prevention[1].

The expansion of the MgO cement paste is caused by the generation and development of magnesium hydroxide crystal.The inflation depends on the amount,shape and size of magnesium hydroxide crystal,the expansion comes from absorbing swelling force and crystallization growth pressure of the magnesium hydroxide crystal[2].The MgO of micro expansive concrete has two sources: the first is contained in cement clinker (containing MgO,the other comes from cement or concrete mixed wiht MgO (the additive MgO).The additive MgO commonly known as "lightburned MgO style" in general under clinker sintering temperature of 800 ~ 1000˚C calcination temperature scope is faster to turns into brucite and is easy to control inflation through adjusting dosage.Comparing with containing MgO,The additive MgO has incomparable advantages,so it is the main research direction of MgO micro expansive concrete.Studies have shown that the content of 5%MgO with calcination temperature 900 ~ 950˚C and size 300 ~ 1180μm had a good compensation effect on hydraulic mass concrete temperature control and crack prevention, but the research has not been tested in the project[3].

The demand of hydraulic mass concrete temperature control and crack prevention are higher and higher with the development of hydropower projects in the west of China.Now 20℃ is defined as a standard curing temperature in general research[4], and various performance tests are carried out accordingly at 20℃.But the rising of temperature is lagre and cooling is slow of the mass concrete inside,external water is difficult to enter.All these are similar to the adiabatic wet environment.The generation rate,position and morphology of concrete expansive sources in this environment have big change and a great influence on development of strength of concrete[5].A large number of engineering practice also had proved that similar MgO have different deformation features in different using conditions of micro expansive concrete.In this article, the effects of different curing temperature on the expansion properties of concrete mixed with magnesium oxide (MgO) were studied by testing the free expansion ratio of cement mortar and the hydration mechanism.

Experiment

Raw materials and mix ratio

Huaxin Dongchuan 42.5 moderate-heat portland cement, Qujing grade I fly ash,and magnesium oxide whose activity indexes were 100s (the required time from magnesium oxide added to a certain concentration of citric acid solution to citric acid solution showing neutral is known for activity index; the higher the activity index, the reactivity of magnesium oxide smaller) were used to do the test. The chemical compositions of the cement, fly ash and light-burned magnesia are shown in Table 1. The particle grade of light-burned magnesia is shown in Figure 1.Basalt artificial sand aggregate was adopted according to following metrics: sand fineness modulus of 2.89, the apparent density of 2890 kg/m^3 gravel crushing index by 3.41%, the apparent density of 2920 kg/m^3.Jiangsu Bote PCA-1 polycarboxylate high performance water reducergent were used as admixtures.

Table 1 Chemical composition of materials w%

Material	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O_{eq}	LOSS
C	60.3	21.7	4.3	4.8	4.9	1.9	0.4	1.9
FA	1.28	53.88	25.41	7.65	3.19	0.73	0.93	3.35
MgO	1.46	0.62	—	0.43	94.28	—	—	—

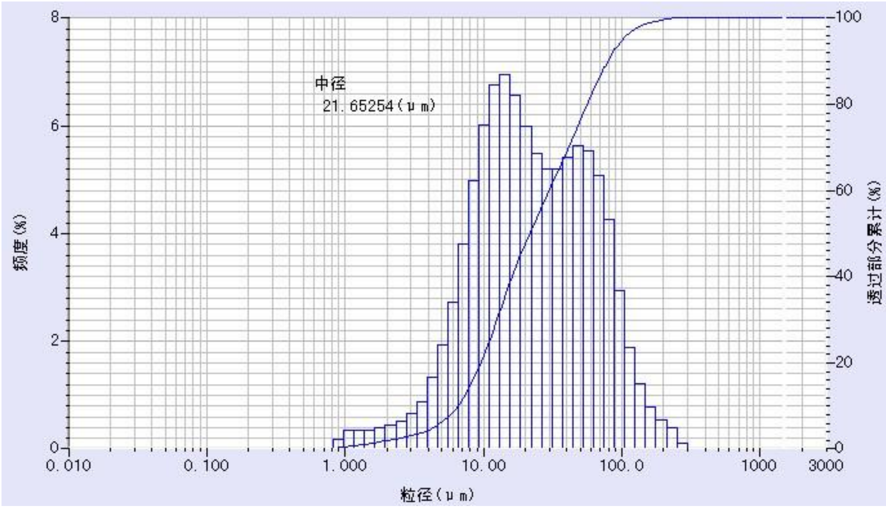


Fig. 1 Particle grade of MgO

Sample preparation and methods.

The mortar free expansion rate test conducted in accordance with the JC453-2004:Method of physical test for self-stressing cement. Light-burned magnesia whose activity indexes were100s was selected to mold mortar; the specimens were demolded to measure the baseline length after curing 24h. Specimens were respectively placed in 20˚C, 30˚C, 80˚C water curing, measuring 3d, 7d, 14d, 28d, 90d, 180d, 240d length changes. To determine the length of the specimen under different curing temperatures, the specimens should be taken out from the curing water before testing the length and put into the wet towel at room temperature of 20˚C (a constant moisture and temperature environment) for 4h to be measured the length.

At the same time, the corresponding gelled material small pieces were molded. The specimens were placed in the same curing condition to the prescribed age for being soaked in anhydrous ethanol. The specimens grinded were analyzed by SEM and TG/DSC microcosmic test.

Results and analysis

Free expansion rate of mortar

The free expansion rate test results of three kinds of curing temperature are shown in Figure 2. The free expansion rate of cement mortar increased with increasing of the content of MgO,and MgO mixed offseted some shrinkage of mortar in the early age.The free expansion rate of specimens increased with temperatures rising. When the MgO cement mortars were cured temperature from 20˚C rose to 80˚C, the cement mortar expansion rate increased quickly at early stage and was slower at late stage. At hydration age 3d,the free expansion rate of cement mortars mixed with 8% MgO were more than 500×10^{-6}, which were equal or more than 40% of inflation rate at hydration age 240d.This showed that with the increase of curing temperature, hydration rate of MgO had a some degree of increasement and hydration completely were earlier.So the expansion curve was sooner to stabilize. The time of mortar expansion rate stabilizing were approximately 28 under 80˚C water curing conditions. The higher content of MgO, mortar free expansion rate were more sensitive to curing temperature.

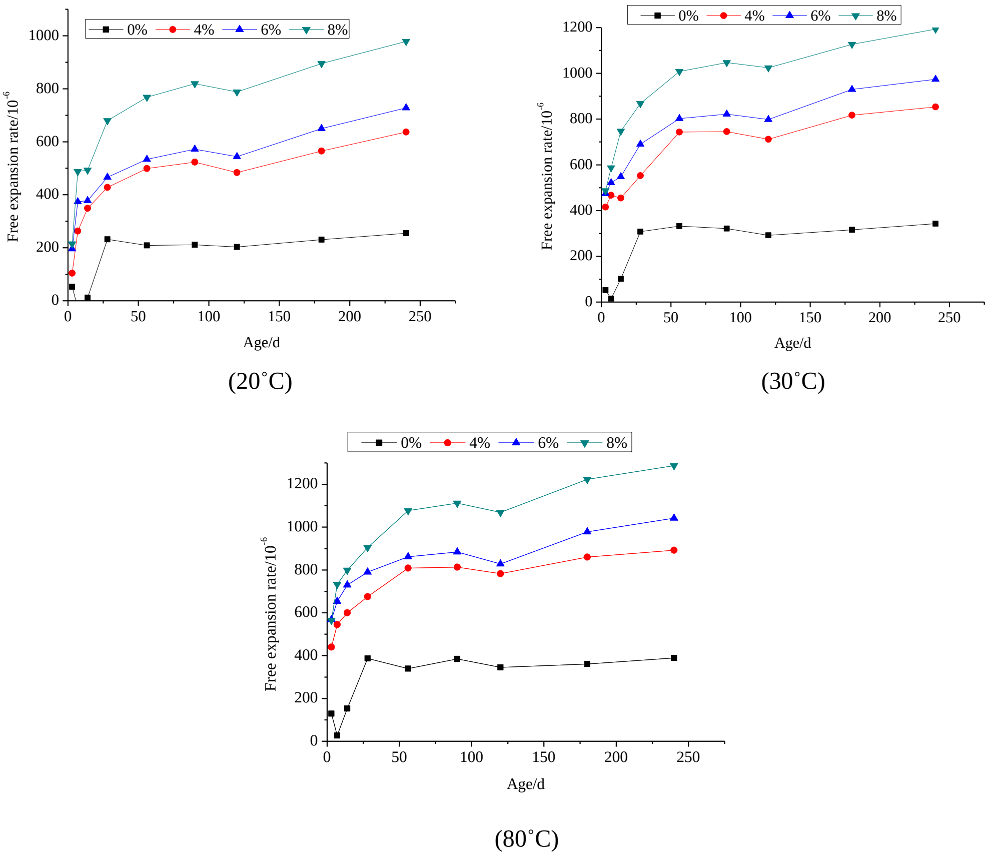
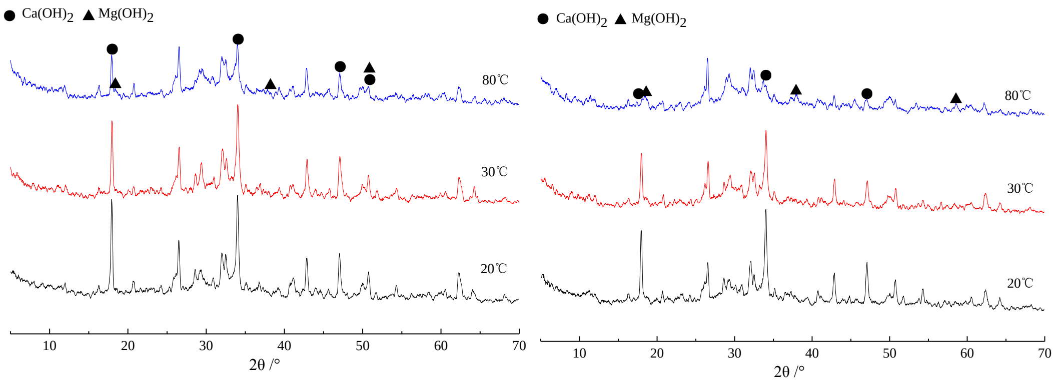


Fig. 2 Time-dependent free expansion rate of mortar at different temperatures

Hydration of MgO in cement paste

The content changes of calcium hydroxide (CH) and magnesium hydroxide ($Mg(OH)_2$) in hardened paste mixed with 6% of MgO were analyzed by XRD. The XRD patterns of three kinds of curing temperature are shown Figure 3.

The results showed that within the hydration age 7d,the content of calcium hydroxide (CH) in hardened paste at 38˚C and at 80˚C water curing conditions were significantly less than the content at 20˚C water curing conditions,due to the rising of curing temperature accelerated the secondary hydration of fly ash and consumed CH,a small amount of MgO had hydrated into $Mg(OH)_2$ in hardened paste at 38˚C and at 80˚C water curing conditions,but there were no $Mg(OH)_2$ generated in hardened paste at 20˚C water curing conditions.Within the hydration age 90d, the MgO were not discovered in the cement paste at 38˚C and at 80˚C water curing conditions, which meaned that the MgO had completely hydrated into $Mg(OH)_2$.Then only a small amount of $Mg(OH)_2$ were discovered at 20˚C water curing conditions.The test results indicated that higher curing temperature promoted the hydration of MgO, the faster hydration.

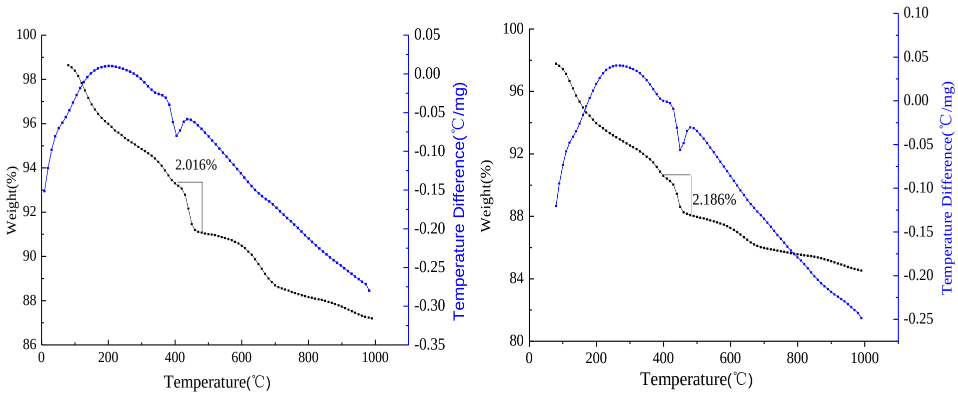


7d 90d

Fig. 3 XRD patterns of pastes with 6% MgO cured at different temperatures

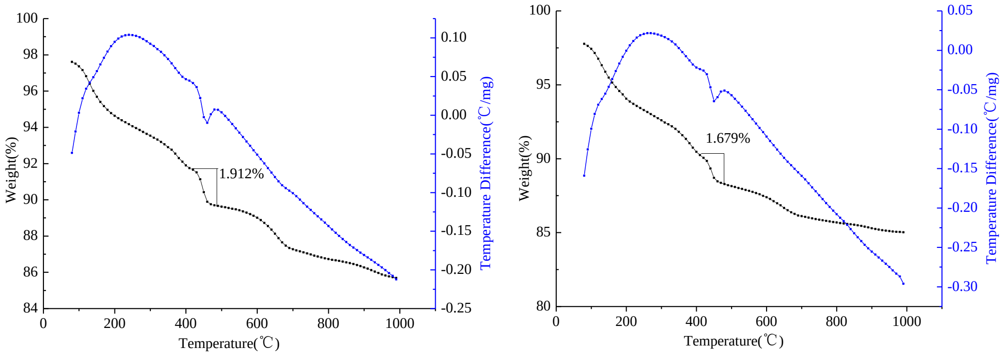
The hardened paste mixed with 6% of MgO were analyzed by TG-DSC,the TG-DSC curves of of pastes of three kinds of curing temperature are shown Figure 4~ Figure 6.

The results showed that the weightlessness of hardened paste mainly concentrated in the 300~ 450˚C temperature range which is the temperature range of CH pyrolysis.But this temperature range was similar to the temperature range of $Mg(OH)_2$ pyrolysis.According to the results of XRD analysis,the weightlessness steps of hardened paste at 20˚C water curing condition were caused by hydroxyl of CH removed.But at 30˚C and 80˚Cwater curing conditions,the weightlessness steps were caused by the hydroxyl of CH and $Mg(OH)_2$ removed.Instead of increasing,the weightlessness reduced with the rising of curing temperature.Because the rising of curing temperature accelerated the secondary hydration of fly ash and the amount of CH reduced.Within the hydration age 90d,the weightlessness steps of different curing temperature were caused by hydroxyl of CH and $Mg(OH)_2$ removed, on the one hand the weightlessness change of hardened paste at 20˚C water curing condition was litter,on the other hand the weightlessness at 30℃ and 80℃water curing conditions reduced.



（a）7d （b）90d

Fig. 4 TG-DSC curves of of pastes with 6% MgO cured at



（a）7d　　　　　　　　　　（b）90d

Fig. 5　TG-DSC curves of of pastes with 6% MgO cured at 30˚C

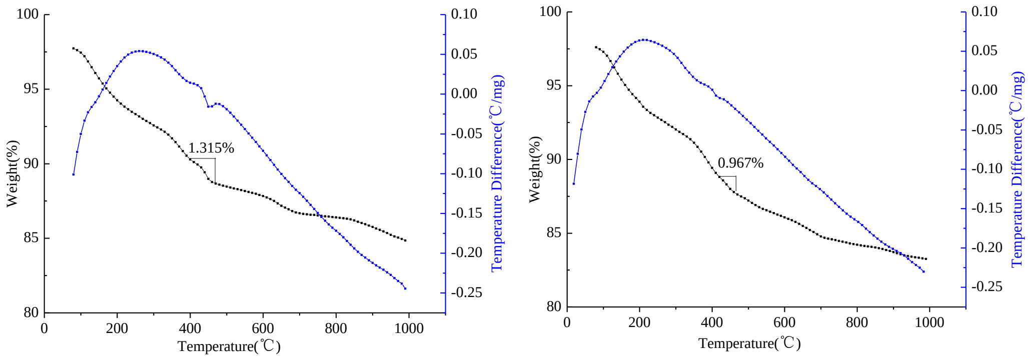
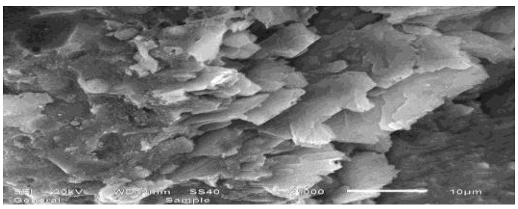
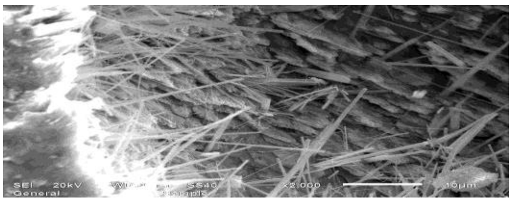


Fig. 6　TG-DSC curves of of pastes with 6% MgO cured at 80˚C

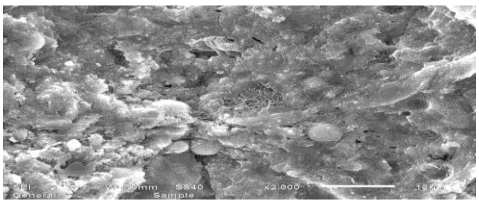
The SEM images of pastes with 6% MgO of three kinds of curing temperature,within the hydration age 90d,are shown were shown Figure7.Some small $Mg(OH)_2$ particles and thin sheet of $Mg(OH)_2$ could be observed in cement paste at 20˚C water curing condition,.At 30 ˚C water curing condition,the hydration products of MgO were acicular crystal, about 0.4 microns long, and distributed in the cement hydration products,so the expansion stress produced by hydration of MgO could be relaxed and the deformation of the concrete was reduced.All these is good for improving the durability of concrete.Under 80˚C water curing condition,the interface of hydration products were close-grained,and pores reduced significantly with micro cracks existing.Because the rising of curing temperature,the crystals of $Mg(OH)_2$ gathered themselves together quickly,which caused crack by the swelling stress[6].



（a）20˚C



（b）30˚C



（c）80˚C

Fig. 7　SEM images of pastes with 6% MgO cured at 20˚C、30˚Cand 80˚C

Conclusions

The research results showed that the free expansion ratios increased different with the rising of curing temperature,the higher the curing temperature, and the earlier expansion to reach stability.With the rising of curing temperature,the extent of MgO hydration improved and the crystals of $Mg(OH)_2$ grow.When curing temperature from 20˚C rose to 30˚C,short lamellar crystals of $Mg(OH)_2$ turned into fibrous and interlaced with each othe.The hydration products gathered themselves together and micro cracks generated at 80˚C curing temperature.The rising of curing temperature accelerated migration rate of Mg^{2+} and OH^- ions after the MgO hydrating, which made the formation of $Mg(OH)_2$ crystal to be accelerated accordingly,and swelling effect in advance.

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