ABNORMAL EXPANSION OF COARSE-GRAINED CALCITE IN THE AUTOCLAVE METHOD

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ABSTRACT

Abnormal expansion of coarse-grained calcite at high temperature (150 ℃) was investigated in this research. Results show that coarse-grained calcite can undergo heat-induced expansion and the expansion value is proportional to the aggregate size. The heat-induced expansion has a negligible effect on the autoclave method identifying of ASR reactivity of aggregates. It is suggested that the expansion of carbonate aggregates in the autoclave method be appraised with the help of other methods, such as petrography.

Key words: Coarse-Grained Calcite, Alkali Reactivity, Autoclave Method

INTRODUCTION

Alkali-aggregate reaction (AAR) includes alkali-silica reaction (ASR) and alkali-carbonate reaction (ACR). Identification of the alkali reactivity of aggregates is important to prevent AAR and to determine whether AAR is the cause of concrete degradation. In ASR research, Ranc et al concluded that ASTM methods C227 and C289 were not reliable in detecting six deleteriously expansive coarse aggregates (Ranc et al.1994). Tang et al proposed an autoclave method for the identification of ASR reactive aggregates, which adopts a high temperature (150℃) and a high alkali content (1.5% Na_2O in specimens and autoclaved in 10% KOH solution) (Tang et al.1983). Inter-laboratory research in France has proved that the microbar method (P 18-588) which adopts 150 ℃ × 6h cure and the autoclave test (P 18-589) which adopts 127 ℃ × 18h cure lead to similar conclusions as to the reactivity or non-reactivity of most of the tested aggregates (Criaud et al.1994).

The most commonly adopted methods in the research of alkali carbonate reaction are ASTM C 1105 and CSA A 23.3-14A, which take a long time and do not detect late-expansive aggregates. It is necessary to develop rapid and effective methods. Based on autoclave method to detect ASR reactivity of aggregates, Tang changed the aggregate size and applied the method to determine the alkali reactivity of carbonate aggregates (Tang et al.1994). Preliminary research has proved that the method can distinguish between safe and harmful carbonate aggregates (Tong 1994).

In the case of ASR, the elevation of temperature only accelerates the reaction process and the corresponding expansion and thus makes it possible to obtain the final result in a shorter period. Therefore, high temperature has been employed in rapid methods to detect the ASR reactivity of aggregates. However, in the case of ACR, both alkali-dolomite reaction and alkali-calcite reaction are promoted at high temperatures in KOH solution. It is not difficult to understand that the rate of ion diffusion and product formation will be accelerated by the elevation of temperature in view of the dynamics. It is also noted that the ion product constant of water K_{H2O} changes at different temperatures, e.g., $K_{H2O,\ 298K}=10^{-14}$, $K_{H2O,\ 348K}=10^{-12.72}$ and $K_{H2O,\ 423K}=10^{-11.64}$. The ion product of water is related to H^+ and OH^- concentration in the solution. Therefore, the alkali-calcite reaction accelerates as the temperature rises. In recent research, it was found that coarse-grained calcite resulted in abnormal expansion of specimens at high temperature (Mu et al.1995). Therefore, attention was given to factors affecting the expansion behaviour and their possible effect on autoclave methods.

EXPERIMENTAL

Three kinds of calcite rocks were used in the research. L1 and L2 are two fine-grained calcite rocks from Nanjing. TS is coarse-grained calcite rock from Tangshan, Nanjing. To understand the performance of coarse-grained calcite at high temperatures, both in the rapid method for ASR and in the rapid method for ACR, TS aggregates were crushed to various groups of different sizes, from 0.15 to 10 mm. Parameters adopted in the experiments were the same as in the literature.

Table 1 Calcite aggregate sizes adopted in experiment

Name	TS1	TS2	TS3	TS4	TS5
Size range (mm)	5.0-10.0	2.5-5.0	1.25-2.5	0.80-1.25	0.15-0.80

RESULTS AND DISCUSSIONS

It is known that limestone is relatively inert aggregate which is widely used in concrete construction. In the present research, TS1, L1 and L2 were used as aggregates in the concrete microbar test to compare their performance at high temperatures.

It is shown in Fig. 1 that TS can result in expansion well above that of L1 and L2 in the concrete microbar test. The expansion value of 0.08% for TS1 after 6h is close to the threshold value (0.1%) for alkali-reactive carbonate aggregate in the concrete microbar test. To understand the effect of heat treatment on the abnormal expansion by TS, TS1 was used as an aggregate in the concrete microbar test with the same total autoclave time (60h) and various time periods (15h, 20h, 30h, 60h). Various heat treatment procedures thus can be expressed in terms of autoclave steps e.g. 1, 2, 3, 4 steps with a fixed toatl autoclave time. The are shown in Fig. 2.

Fig. 2 shows that the heat treatment procedure has a profound effect on the expansion behaviour of TS1 aggregate in concrete microbar test. With equal total autoclave time, the more the steps of a heat treatment procedure, the higher the expansion value. Since the total autoclave time is fixed at 60h, the chemical reaction involved can be regarded as the same. Therefore, the differences in expansion result from the different approaches of autoclave. In a control experiment with the same procedure, alkali-reactive aggregate from Canada (CK) was employed. The result is shown in Fig. 3. From Fig. 2 and Fig. 3, it is appropriate to consider the abnormal expansion caused by coarse-grained calcite as heat-induced expansion.

A series of experiments were conducted to evaluate the effect of aggregate size. The results under various conditions are shown in Fig. 4, Fig. 5 and Fig. 6. It is evident that the expansion caused by TS aggregate is closely related to the aggregate size. The expansion caused by TS aggregate is similar when autoclaved in alkali solution, water and vapour respectively. The larger the aggregate size, the higher the expansion value.

When cured in alkali solution and water at identical grain sizes, the average expansion values are wholly higher than those in vapour. The possible reason is that alkali solution and water curing can make it easier for the chemical reaction to proceed. The expansion values in alkali solution and water are very close, which may suggest that water curing is perhaps sufficient for the chemical reaction and external alkali provision has little effect on the expansion. This phenomenon may suggest that the alkali ions play different roles here compared with their roles in the expansion process resulting from alkali-silica reaction. In previous research, XRD proved that $Ca(OH)_2$ formed in the cleavages of calcite aggregates, which facilitated the expansion. To determine the effect of chemical reaction on expansion, an experiment was carried out to determine the expansion of TS in different alkali environments in the concrete microbar method.

Table 2 Expansion of specimens containing TS1 aggregate in various alkali environments

Samples	TS1	TS1P*	TS1W**
Alkali content ($Na_2O_{equiv.}$) in specimens (%)	1.5	0.46	0.23
Immersion solution	10% KOH a.q.	H_2O	H_2O
Expansion after 6h (%)	0.09	0.087	0.082

* *TS1P: Ordinary portland cement was used.*

** *TS1W: White cement with low alkali content was used.*

Table 2 shows that alkali-calcite reaction has a limited effect on the specimen expansion. This is in accordance with the conclusion on heat-induced expansion above.

It is noted that the difference between the rapid mortar bar method for ASR and the concrete microbar rapid method for ACR is in the aggregate sizes adopted, with the former of 0.15-0.80 mm and the latter of 5.0-10.0 mm. Experiments were done to compare the expansion behaviour of calcite aggregate in both autoclave methods. The results in Table 3 show that the mortar bar method for detecting ASR reactivity is not affected by the abnormal heat induced expansion of coarse-grained calcite.

Table 3 Expansion of TS calcite aggregate in autoclave methods

Name of autoclave method	Concrete microbar method	Mortar bar method
Expansion after 6 h (%)	0.091	0.015

CONCLUSION

The abnormal expansion of coarse-grained calcite aggregate in the concrete microbar test is mainly heat induced and is dependent on the particle size. Expansion resulting from alkali-calcite reaction at high temperatures is very limited, and will not contribute to the alkali reactivity of carbonate aggregates in the concrete microbar test. To get a complete understanding of the expansion of carbonate aggregates in the rapid methods, petrographic examination or control tests with very low alkali content are helpful.

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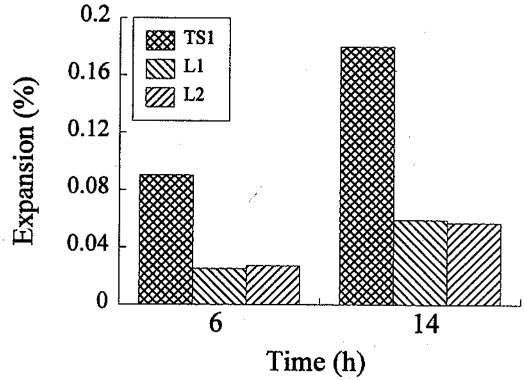

Fig. 1 Comparison of expansion behaviour of different calcite aggregates

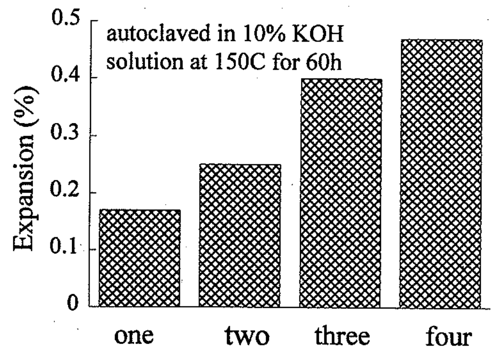

Fig. 2 Effect of various heat treatment procedures on expansion behaviour of TS1 aggregate

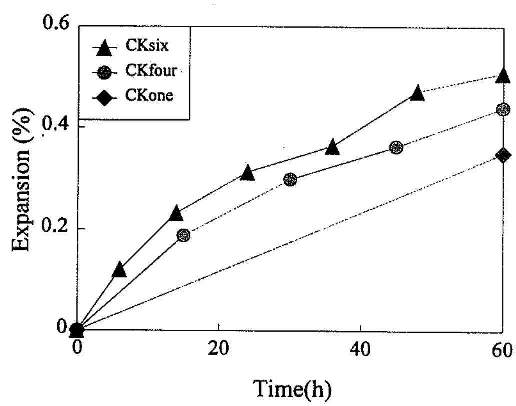

Fig. 3 Effect of various heat treatment procedures on expansion of CK aggregate

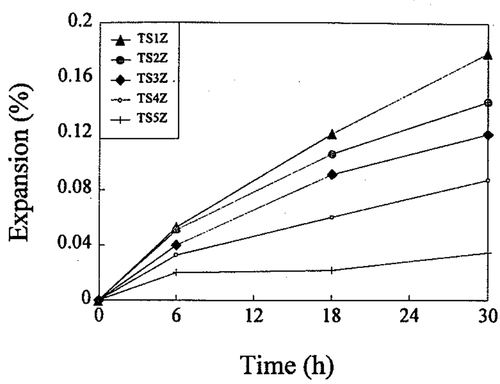

Fig. 4 Expansion of concrete microbars containing TS aggregates cured at 150 ℃ in vapour

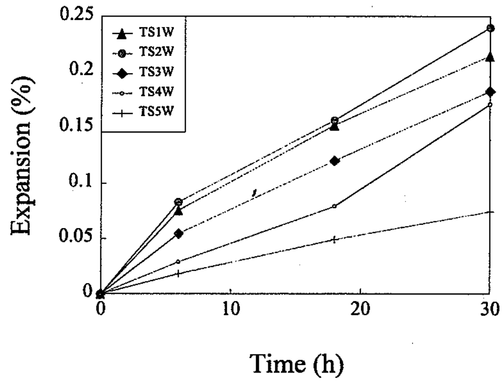

Fig. 5 Expansion of concrete microbars containing TS aggregates cured at 150 ℃ in water

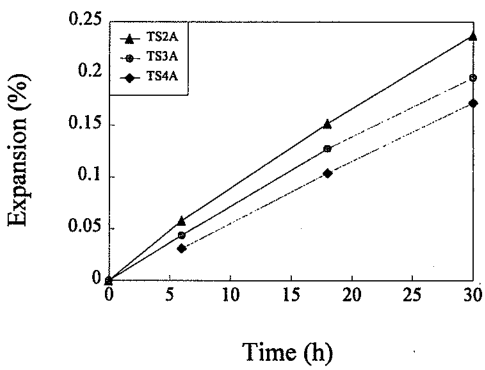

Fig. 6 Expansion of concrete microbars containing TS aggregates cured at 150 ℃ in 10% KOH Solution