Reversal of Setting Expansion of Gypsum-Bonded Investment Containing Admixed $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$

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Received on January 19, 1984

Reversal or loss of setting expansion gypsum-bonded investment is studied as a function of mixing rate and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ content. Investments containing $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ mixed at higher speeds (1000 rpm and 1680 rpm) exhibited at 24 hours reversal or loss of setting expansion which is directly related to both the speeds of mixing and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ content.

Key Words: Investment, Reversal, Setting expansion.

INTRODUCTION

Gypsum-bonded investments expand while setting, and this expansion coupled with subsequent thermal expansion, provides a mold sufficiently enlarged to compensate for the shrinkage of molten alloy upon cooling from casting temperature to room temperature. Previous papers^{1,2)} have reported a reversal or loss of setting expansion over a 24 hr. period under certain conditions of mixing and ring configuration. The degree of reversal was positively correlated with mixing rate and powder/water ratio. Other studies³⁾ have shown this reversal in setting expansion to be related as well to the content of calcium sulfate dihydrate (CSD) added to the investment prior to mixing.

The purpose of this study was to investigate the combined effects of CSD content and mixing rate upon the setting expansion of a gypsum-bonded investment.

MATERIALS AND METHODS

The materials used in this study are listed in Table 1. Shofu cristobalite* investment was shown by X-ray diffraction analysis to be free of CSD. Experimental samples were produced by adding 0, 1, 2 and 3% CSD to 100 g. samples of Shofu cristobalite investment. All samples were prepared as 100 g. of powder and mixed at W/P ratio of 0.35.

The experimental arrangement used to measure axial setting expansion in standard 0.9 mm thick dry asbestos⁺⁺ lined 18:8s.s. casting rings ($\phi 30$ mm, 35 mm length) is shown in Figure 1.

* Shofu, Kyoto, Japan

+ Whip-Mix Corp. Louisville, Ky.

++ Morita Corp. Kyoto, Japan

Table 1 Materials used and Experimental Methods

Investment Used	Shofu cristobalite
W/P ratio	0.35 constant
CaSO ₄ ·2H ₂ O added	0%, 1%, 2% and 3%
Mixing Condition	Hand mixing: 15 sec (120 rpm) Mechanical (1) 450 rpm (2) 1000 rpm 45 sec (3) 1680 rpm

Figure 1 Apparatus for measurement of axial setting expansion

Investment samples were mechanically mixed at 450, 1000 or 1680 rpm. for 45 sec. using a Vac-U-Vester⁺. The rings were placed as illustrated in Figure 1 upon a flat glass plate and filled with mixed investment. The glass plate and filled ring was then placed on the table of an electric dilatometer⁺⁺, and a transducer bar weighing 0.5 g. and 10 mm in diameter was placed 1 mm. deep using caliper into the investment at the center of the ring.

Each setting expansion measurement was started 1 minute after commencement of spatulation, and expansion data were recorded continuously for 24 hrs. at 23°C and 50% relative humidity. Five samples of each mixing condition were measured for each experimental investment, and the data were averaged.

RESULTS AND DISCUSSION

The averaged data are presented in Table II and are graphically illustrated in Figures 2, 3, and 4.

For all samples mixed at 450 rpm: No change from the maximum setting expansion was detected over a 24 hr. period for any concentration of admixed CSD. Increasing the CSD did, however, decrease slightly the time required to attain maximum expansion (T_{E-max}) from 148 minutes for samples containing no CSD to 125 minutes for samples containing 3% CSD (Table II; Figure 2)

For samples mixed at 1000 rpm: The maximum setting expansion (E-max) was maintained at 24 hrs. only for those samples containing no CSD. Increasing the CSD content by 1, 2, and 3% resulted in a reversal of E-max of 0.7, 4.0, and 10.1% respective-

⁺ Whip-Mix Corp. Louisville, Ky.

⁺⁺ Shinko-Denki Corp. Kobe, Japan

Table 2 Expansion data for various $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ contents and mixing speeds

Investment	Mixing speed (rpm)	% $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ added	Time before initial setting expansion (min)	Time to maximum setting expansion (min)	Maximum setting expansion (%)	Setting expansion after 24 hours (%)	% decrease of maximum setting expansion
Shofu Cristobalite Investment Containing no $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	450 rpm	0	7.6 (0.1)	148 (5.1)	1.49 (0.05)	1.49 (0.05)	0
		1	7.8 (0.3)	140 (0.8)	1.42 (0.07)	1.42 (0.07)	0
		2	7.3 (0.2)	130 (2.6)	1.19 (0.12)	1.19 (0.12)	0
		3	7.5 (0.3)	124 (5.5)	1.08 (0.07)	1.08 (0.07)	0
	1000 rpm	0	5.7 (0.1)	127 (2.0)	1.56 (0.11)	1.56 (0.11)	0
		1	5.6 (0.2)	111 (6.6)	1.36 (0.03)	1.35 (0.02)	0.71 (0.63)
		2	5.5 (0.2)	97 (1.8)	1.30 (0.01)	1.24 (0.02)	4.00 (1.77)
		3	4.5 (0.3)	87 (4.2)	1.28 (0.04)	1.15 (0.04)	10.07 (1.71)
	1680 rpm	0	4.5 (0.4)	125 (0)	1.64 (0.05)	1.64 (0.05)	0
		1	3.4 (0.1)	71 (6.6)	1.46 (0.04)	1.26 (0.05)	13.85 (3.29)
		2	2.9 (0.1)	54 (1.0)	1.28 (0.04)	0.72 (0.05)	43.67 (3.55)
		3	2.5 (0.2)	51 (2.4)	1.09 (0.05)	0.32 (0.11)	71.10 (8.44)

Figure 2 Setting expansion of investment mixed at 450 rpm

Figure 3 Reversal of setting expansion of investment mixed at 1000 rpm

Figure 4 Reversal of setting expansion of investment mixed at 1680 rpm

ly. (Table II; Figure 3)

For samples mixed at 1680 rpm: There was no reversal of setting expansion for samples which contained no CSD, but when the CSD concentration was increased to 1, 2, and 3%, the E-max decreased by 13.8, 43.6, and 70.8% respectively. (Table II; Figure 4)

Figure 5 Percent decrease in 24 hour setting expansion as a function of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ content and mixing speed

Figure 6 Effect of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ content and mixing speed on time before detection of initial setting expansion

Figure 5 illustrates the relation between the decrease in E-max over 24 hrs. as a function of CSD concentration for each mixing speed. At all mixing speeds, there was a loss of E-max as the CSD content was increased, the magnitude of loss increasing with increasing mixing speed for a given CSD content.

Figure 6 illustrates the effects of CSD content and mixing speed on the time required to detect initial expansion (T_i). For all investments mixed at 450 rpm., $T_i = 7.5$ min. Increasing the mixing rate decreases T_i for a given CSD concentration, and increasing the CSD concentration also decreases T_i for a given mixing rate. The most rapid set ($T_i = 2.5$ minutes) was achieved in those investments containing 3% added CSD and which were mixed at 1680 rpm.

Figure 7 illustrates the effects of CSD content and mixing speed on the time required to achieve maximum expansion ($T_{E\text{-max}}$). At all mixing speeds, $T_{E\text{-max}}$ decreases with increasing CSD content, and for a given CSD content, $T_{E\text{-max}}$ decreases with increasing mixing speed. This is likely the result of additional nuclei of crystallization produced by more rapid rate of mixing.

Figure 8 shows the correlation the time required to achieve a maximum expansion ($T_{E\text{-max}}$) with the degree of actual percent loss of setting expansion. In logarithm-logarithm, there is a strong correlation between these values. The samples which set most rapidly also showed the greatest loss of setting expansion after 24 hrs.

Figure 7 Effect of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ content and mixing speed on time required to achieve maximum setting expansion

Figure 8 Logarithmic correlation plot of $T_{\eta\text{-max}}$ vs. loss of setting expansion

CONCLUSION

The reversal of the maximum setting expansion of gypsum-bonded investment was observed when CSD was added to the investment and when high speed mixing was employed. The degree of loss of setting expansion appears to be strongly correlated with those factors which lead to rapid set of the investment, although the specific mechanism for loss of expansion is not known.

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本号掲載論文の和文抄録

CaSO₄·2H₂O 添加による石こう系鋳型材の硬化時膨張 の反転現象について

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石こう系鋳型材を 1680 rpm の速度で機械練和した際に発現した膨張が経時的に反転あるいは減少することを発見した。この原因は硬化時間の短縮が影響していると考えられた。

そこで、硬化時間の促進として作用する CaSO₄·2H₂O の添加と混和速度を組合せて硬化時膨張の反転現象を検討した。

最大硬化時膨張の反転は CaSO₄·2H₂O が添加された場合でしかも 1000 rpm および 1680 rpm の高速で混和

されたときに起こる。そしてこの反転率は CaSO₄·2H₂O の量と混和速度に影響される。すなわち硬化時間を促進する要因が硬化時膨張の経時的な反転に関与していることが判明した。3 g の CaSO₄·2H₂O を添加し、1680 rpm で混和したものは 24 時間後には最大硬化時膨張値が 71% 減少していた。一方 3 g の CaSO₄·2H₂O の添加でも 450 rpm での混和では最大硬化時膨張の反転は出現しない。

タンニン・フッ化合物剤 (HY 剤) を配合した 高銅型アマルガムの物理的性質

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タンニン・フッ化合物剤 (HY 剤) 配合単一組成型高銅アマルガムの物理的性質と 12 ヶ月間浸水の及ぼす影響を検討した。フッ化錫配合の削片状低銅型アマルガム (市販品) 並びにフッ化物を含まない削片状低銅型アマルガムと単一組成型高銅アマルガムを対照として用いた。HY 剤の配合が高銅型アマルガムの物理的性質に及ぼす影響についてみると、0.5 wt% までは著明ではないが、1.0 wt% ではクリープを除いて著明であった。水中浸漬後では径方向引張り強さの低下が著明であったが圧縮強さの低下はそうではなく、クリープは変化しなかつ

た。一方、フッ化錫含有削片状低銅型アマルガムは水中浸漬後、物理的性質はいずれも著明に低下した。微細構造の変化は高銅型アマルガムについては含有されていた HY 剤の脱落による空隙の発生以外にはみられなかったが、低銅型アマルガムの方はフッ化錫の脱落による空隙の他に γ_2 相の腐食や溶解の様子がみられ、物理的性質の低下と関連していた。これらの所見は対照として用いたフッ化物を含有しない低銅型アマルガムについても同様であった。