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Effects of MgO-based expansive additive on compensating the shrinkage of cement paste under non-wet curing conditions

L. Mo [a,*], M. Deng [a], A. Wang [a,b]

[a] College of Materials Science and Engineering, Nanjing University of Technology, Nanjing 210009, PR China
[b] Anhui Key Laboratory of Advanced Building Materials, Anhui Institute of Architecture and Industry, Hefei 230022, PR China

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ABSTRACT

Expansive additives are widely used to compensate the drying shrinkage of cement-based materials to avoid cracking. However, the expansion of conventional ettringite-bearing expansive additive depends strongly on wet curing and is mainly generated at early age, and hence it may not work well in concretes without sufficient water supply or exhibit long-term shrinkage. MgO-based expansive additive, for which less water is needed for the formation of $Mg(OH)_2$ in comparison to ettringite, was prepared and its compensating effect on the autogenous shrinkage and late age thermal shrinkage of Portland and fly ash cement pastes at low water-to-cement ratio was investigated. The tests were conducted under sealed condition, so that the moisture exchange with the environment was prevented. Results show that, even under the non-wet curing condition, the shrinkages of cement pastes can be compensated effectively. Microstructure analysis by scanning electron microscope indicates that the macro-expansion of cement pastes is probably caused by the locally restrained expansion of MEA due to the hydration of MgO.

1. Introduction

Volume shrinkages of cement-based materials, such as drying shrinkage, autogenous shrinkage, and thermal shrinkage, may crack the materials and hence negatively influence the durability of concrete structures. For autogenous shrinkage cracking, many different mitigation strategies have been developed [1]. Proper external moist curing is a widely used conventional method to prevent cracking in some situations [1]. However, for high performance concrete with low permeability at low water-to-cement ratio or mass concrete, the conventional external moist curing may be ineffective to prevent the self-desiccation of concrete materials. Recently, internal curing, which provides additional moisture through water saturated porous lightweight aggregate (LWA) or super-absorbent polymers (SAPs), is considered to be a very promising technique for reducing self-desiccation and preventing autogenous shrinkage [2–6]. However, in some cases, the replacement of normal aggregate by LWA has a negative influence on the strength of concrete [7]. Moreover, the super-absorbent polymers may be expensive. In order to reduce the drying shrinkage of concrete, shrinkage-reducing admixtures have been commonly used [8,9] and even coupled with expansive agents [10,11].

The expansion generated by the hydration of expansive components in cement, such as sulfo-aluminate, free calcium oxide and uncombined magnesia (MgO), was usually used to compensate the shrinkage of cement-based materials. In order to adjust the mixing content of expansive components in concrete conveniently, the expansive components were produced separately from cement and used as additives, and thus various types of expansive additive were developed [12–14]. However, as a large amount of water is needed for the formation of ettringite, the ettringite-bearing expansive additive (i.e. sulfo-aluminate based expansive additive) depends strongly on wet curing. Usually a continuous wet curing of at least 7 d after casting was recommended for concrete containing the ettringite-bearing expansive additive to generate sufficient expansion [12]. Therefore, the application of this kind of large water-consuming expansive additive may be ineffective in concrete with limited water curing. In addition, for the high performance concrete with low permeability or mass concrete, the exterior water may have difficult penetrating into the concrete, and thus insufficient water may be supplied for the complete hydration of the large water-consuming expansive additive even if the exterior wet curing is provided.

For mass concrete, besides the autogenous shrinkage, thermal shrinkage at later age plays an important role in concrete cracking. Due to the heat liberation of cement hydration and low thermal conductivity of concrete, a high temperature rise may be caused. Therefore, large thermal shrinkage may be induced during the cooling stage, resulting in excessive thermal stress and even cracking the mass concrete. Control of the temperature rise in mass concrete is a widely used conventional method to prevent thermal

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[*] Corresponding author. Tel.: +86 25 83587218.
E-mail address: andymoliwu@sohu.com (L. Mo).

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cracking of mass concrete, and this method includes using low-heat materials, pre-cooling or post-cooling concrete, and surface insulation [15]. Some of these traditional temperature controlling methods, for example, the cooling of concrete, are expensive and time consuming. Furthermore, for mass concrete applications with higher temperature rise and longer cooling age, i.e. the dam concrete in which the cooling age may last several months or even years, it is difficult to completely prevent the thermal cracking by only taking the temperature controlling methods [16].

Compensating the thermal shrinkage may be an effective approach to prevent thermal cracking of mass concrete. However, the expansion of the ettringite-bearing expansive additive is mainly generated at early age, within 14 d, which is earlier than the cooling age of mass concrete, so it may be inadequate for compensating the long term thermal shrinkage of mass concrete. Mehta et al. [17] proposed to produce self-stress in mass concrete by using MgO additive to prevent thermal cracking. In the 1970s, it was reported that the thermal cracking of dam concrete in Baishan hydropower station was prevented due to the compensation effects of a delayed expansion on the thermal shrinkage of mass concrete at late age [16]. The delayed expansion was produced by the slow hydration of MgO contained in cement with high MgO content [16]. Since then, much attention has been attracted on the compensating effects of MgO contained in cement on thermal shrinkage of mass concrete in China. However, the MgO contained in cement clinker is dead burnt and its content is usually limited for the sake of cement soundness. In order to avoid the dead burnt of MgO and precisely control its content and hydration activity, MgO-based expansive additive (MEA) has been produced by calcining magnesite or other Mg-bearing minerals separately from the production of cement [16,18]. Our recent research proved that the hydration activity and expansion property of MEA could be designed by regulating the calcining temperature and residence time [19].

With the development of high performance concrete with low water-to-cement ratio, a denser microstructure may be formed, which may prevent the introduction of exterior water into the inner part of mass concrete for the complete hydration of cement and additives. How does the MEA work in cement-based materials at low water-to-cement ratio and without exterior wet curing? Few works have been focused on this topic. This study aims to investigate the hydration of MEA and its effects on compensating the shrinkage of cement paste with low water-to-cement ratio under the non-wet curing condition provided by sealing the samples and preventing the moisture exchange with the environment.

2. Experimental procedures

2.1. Materials

Portland cement was prepared by inter-grinding cement clinker with 5% of gypsum (by weight of cement clinker). The cement clinker contains 70.5% C_3S, 9.6% C_2S, 7.8% C_3A, 10.6% C_4AF, and 0.3% free CaO. Fly ash cement was produced by replacing 30% by weight of cement clinker with type II fly ash. The chemical compositions of cement clinker and fly ash are shown in Table 1.

A type of MEA produced by calcining magnesite in a shaft furnace at about 1100 °C was used. The MEA was then ground into a powder passing 0.08 mm sieve with a residue of 6%. As shown in Fig. 1, the MEA mainly consists of magnesia (MgO), and the MgO content is 91.96% (Table 1). For comparison, a type of aluminate-based expansive additive (noted as AEA, of which the main expansive hydration product is ettringite) produced by JiangSu Bote New Materials Corp. was used. According to the XRD analysis of AEA as shown in Fig. 2, its main expansive ingredients are calcium aluminate oxide (C_3A, C_3A_5) and anhydrite ($CaSO_4$). Its chemical compositions are also shown in Table 1.

2.2. Apparatus

2.2.1. Cylinder mould

Polyethene cylinders with a size of ø150 mm × 250 mm were prepared, of which the bottoms were sealed tightly to avoid moisture loss of cement paste (Fig. 3).

2.2.2. Strain transducer

The deformation of cement paste was test by Type-VWS strain transducer with a length of 100 mm made by GN Instrument Corp., Nanjing, China. The strain transducer deforms simultaneously and equivalently with the cement paste. The total strain of specimen was calculated by Eq. (1), and the autogenous deformation of cement pastes could be obtained by removing the thermal deformation according to Eq. (2):

$$S_{total} = k^{*}\Delta F + b * \Delta T = k^{*}(F - F_0) + (T - T_0) \tag{1}$$

$$S_{auto} = k^{*}\Delta F + (b - a)^{*}\Delta T = k^{*}(F - F_0)^{*}(T - T_0) \tag{2}$$

where S_{total} is the total strain of cement paste; S_{auto} is the autogenous deformation of cement paste; k is a constant of the strain transducer; ΔF is the variation of vibrating modulus, which varies with the length changing of the strain transducer induced by the deformation of cement paste; ΔT is the temperature variation; b is temperature factor of the strain transducer, T_0 and T represent the temperatures collected at the reference time and test time respectively; F_0 and F represent the vibrating modulus of the transducer at the reference time and test time respectively; a is the linear thermal expansion coefficient of cement paste, which was test via increasing the temperature of cement pastes from 25 °C to 50 °C and the average value 16.6×10^{-6}/°C was taken.

2.3. Preparation of cement pastes

Two addition levels of 5% and 8% of MEA by weight of cement were used for both of the Portland cement and fly ash cement pastes. For comparison, Portland cement pastes containing 5% and 8% of AEA (by weight of cement) were also prepared

Table 1
Chemical compositions of raw materials.

	Chemical components (%)								
	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	K_2O	Na_2O	LOSS
Cement clinker	66.13	20.66	4.75	2.88	2.69	1.04	0.43	0.36	1.06
Fly ash	6.12	46.83	36.44	2.95	1.86	1.37	0.63	0.56	3.24
Gypsum	24.51	16.52	1.58	0.61	36.17	2.31	–	–	17.52
AEA	25.36	20.14	9.14	1.66	31.97	2.63	–	–	7.82
MEA	1.68	2.11	0.20	0.66	0.04	91.96	–	–	2.93

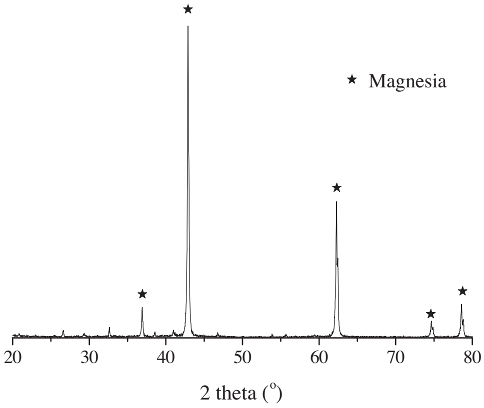


Fig. 1. X-ray diffraction patterns of MgO-based expansive additive.

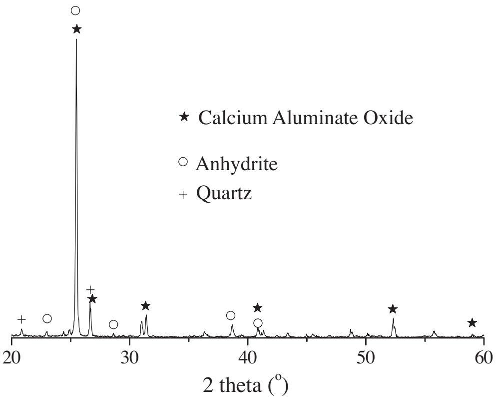


Fig. 2. X-ray diffraction patterns of aluminate-based expansive additive.

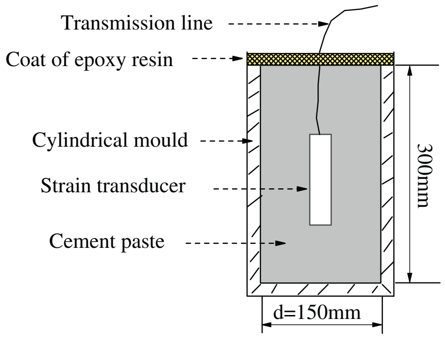


Fig. 3. Schematic representation of the mould and cement paste specimen.

respectively. In addition, for each kind of cement, reference cement pastes without addition of MEA or AEA were cast.

Expansive additives were first mixed into cement homogenously, and then the water at a w/c ratio of 0.28 was added into the cement mixtures and mixed for 4 min to obtain consistent mixtures. The consistent mixtures were cast into the prepared polyethene moulds. The strain transducer was embedded vertically into the centre of the cylindrical paste during the casting process as shown in Fig. 3. The cement pastes were compacted by vibrating for 1 min on a vibrating table. Subsequently, the cylindrical pastes were immediately coated by epoxy resin, so that the moisture exchange with the environment was prevented. Then the cement specimens were placed in the curing box. This study focuses on the deformation of cement pastes after 24 h, so the reference data, namely T_0 and F_0, were collected at 24 h after the casting of cement paste.

All the cement pastes were put into curing boxes in which the temperature could be controlled to simulate a realistic temperature history of mass concrete in field. All the cement pastes underwent a similar temperature development process, reaching the maximum temperature of approximately 44 °C around 16 d and then declining.

2.4. Microstructure characterisation

In order to investigate the hydration characteristic of MEA in Portland cement paste and fly ash cement paste, fractured surfaces of cement pastes at age of 540 d were investigated by Scanning Electron Microscope (SEM, JSM-5900, JEOL, Ltd., Japan). Polished samples were prepared according to the usual sample preparing process of backscatter SEM examination (BSEM) [20] and then investigated by BSEM.

3. Results and discussion

3.1. Autogenous deformation of cement pastes

Fig. 4 shows the autogenous deformations of Portland cement pastes containing various contents of MEA under the non-wet curing condition. The reference Portland cement paste without addition of MEA shrank dramatically at early age, which was attributed to the autogenous shrinkage due to the hydration of cement. A knee-point on the autogenous shrinkage curve occurred at 20 d, and then the shrinkage curve developed slowly. At 30 d, the shrinkage was approximately -320×10^{-6} and the shrinkage curve of cement paste reached a plateau gradually. Compared to the reference Portland cement paste, small increases in the shrinkages were observed in the cement pastes containing 5% and 8% of AEA. For AEA, a large volume of water is needed for the formation of ettringite as described in the following equation:

$$C_3A + 3CaSO_4 + 32H_2O \rightarrow C_3A3CaSO_432H_2O \quad (3)$$

Weight: 270 408 576.

Water/AEA ratio: 576/(270 + 408) = 0.85.

However, a low water-to-cement ratio of 0.28 was used in the cement pastes, so that insufficient intrinsic water may be provided for the complete hydration of AEA to produce sufficient expansion to compensate the shrinkage of cement pastes.

For cement pastes containing MEA, obvious shrinkages also occurred at early age. Due to the slow hydration of MgO in MEA, the expansion produced at early age was smaller than the autogenous shrinkage induced by the hydration of cement simultaneously, and thus only partial autogenous shrinkage was compensated. However, as a result of the compensation effect of MEA, the maximum

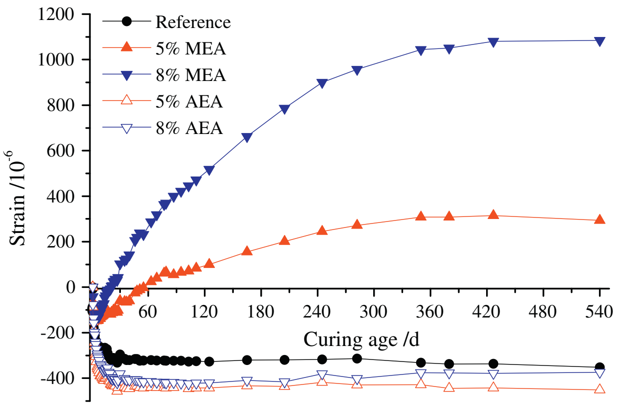


Fig. 4. Autogenous deformations of Portland cement pastes containing various contents of MEA under non-wet curing condition.

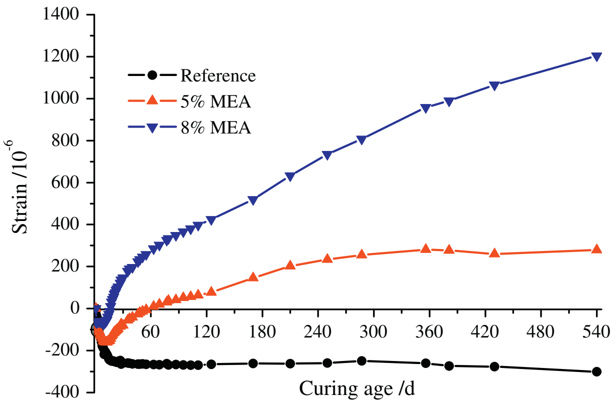


Fig. 5. Autogenous deformations of fly ash cement pastes containing various contents of MEA under non-wet curing condition.

Table 2
Thermal deformations, autogenous deformations, and total deformations of cement pastes during the cooling stage (from the time of peak high temperature to 540 d).

MEA content (%)	Portland cement pastes (10^{-6})			Fly ash cement pastes (10^{-6})		
	Thermal	Autogenous	Total	Thermal	Autogenous	Total
0	−279	−46	−325	−297	−19	−316
5	−279	408	129	−297	447	150
8	−279	1098	819	−297	1193	896

shrinkages of the Portland cement pastes decreased. For example, as illustrated in Fig. 4, the maximum shrinkage of cement paste containing 5% of MEA was -143×10^{-6}. With the continuous hydration of MgO, the shrinkage of cement paste was gradually compensated by the expansion produced by MgO. Shorter time was needed for the complete compensation of the autogenous shrinkage of cement paste containing higher content of MEA. For example, the cement paste containing 8% of MEA showed expansion after 21 d, while cement paste containing 5% of MEA began to expand after 55 d (Fig. 4). At 380 d, the expansions of Portland cement pastes containing 5% and 8% of MEA were 308×10^{-6} and 1050×10^{-6} respectively, and after then, the expansions curves gradually leveled off (Fig. 4).

Fig. 5 shows the autogenous deformations of fly ash cement pastes containing various contents of MEA under the non-wet curing condition. Reference fly ash cement paste showed dramatic shrinkage at early age. A knee-point occurred in the deformation curve of the reference fly ash cement paste at 30 d, being later than 20 d of the reference Portland cement paste (Fig. 4). This may be associated with the slower hydration of fly ash cement in comparison to the Portland cement, which induced slower autogenous shrinkage. The shrinkage of the reference fly ash cement paste at 30 d was -260×10^{-6}, being less than -320×10^{-6} of the reference Portland cement paste at the same age. Due to the expansion produced by MEA, the maximum autogenous shrinkages of the fly ash cement pastes containing 5% and 8% of MEA at early age were decreased to -160×10^{-6} and -70×10^{-6} respectively. And the shrinkages of fly ash cement pastes containing 5% and 8% of MEA were compensated completely at approximately 60 d and 16 d respectively. At 540 d, the expansion of fly ash cement paste containing 8% of MEA was 1203×10^{-6}, being larger than 1085×10^{-6} of the Portland cement paste containing the same content of MEA. Moreover, the expansion seems to continue further. Some studies reported that the fly ash had an inhibition effect on the expansion of MgO, and the inhibition effect was considered to be related to the decrease of alkali environment and porous microstructures formed in cement pastes due to the incorporation of fly ash [21,22]. However, long-term deformation results in this study show that the fly ash seems to delay the expansion of MgO rather than suppress it. Accordingly, the autogenous shrinkage of blended cement pastes containing fly ash can also be compensated effectively by MEA.

3.2. Effects of MEA on compensating the thermal shrinkage

Fig. 6 shows the total deformations of Portland cement pastes containing various contents of MEA undergoing a simulated temperature development process of mass concrete. The temperature increased fast at early age and then declined gradually after 16 d. The total deformation of the reference Portland cement paste was influenced by both of the autogenous shrinkage caused by cement hydration and thermal deformation related to the temperature variation. As seen in Fig. 6, with the increasing temperature, the pastes showed gentle expansion. This may be ascribed to that the thermal expansion caused by temperature rise exceeded the autogenous shrinkage generated simultaneously. At the cooling stage, the pastes shrank quickly with the decreasing temperature. For Portland cement pastes containing MEA, besides the autogenous shrinkage and thermal deformation, the expansion caused by the hydration of MgO also contributed to the total deformation. The total deformation curves of cement pastes containing MEA

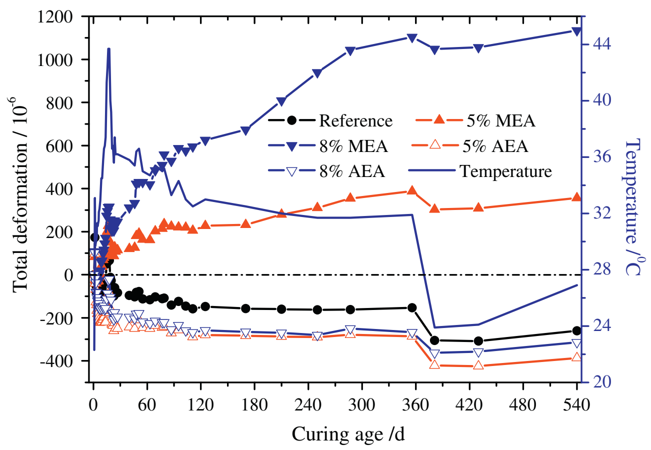


Fig. 6. Total deformations of Portland cement pastes containing various contents of MEA.

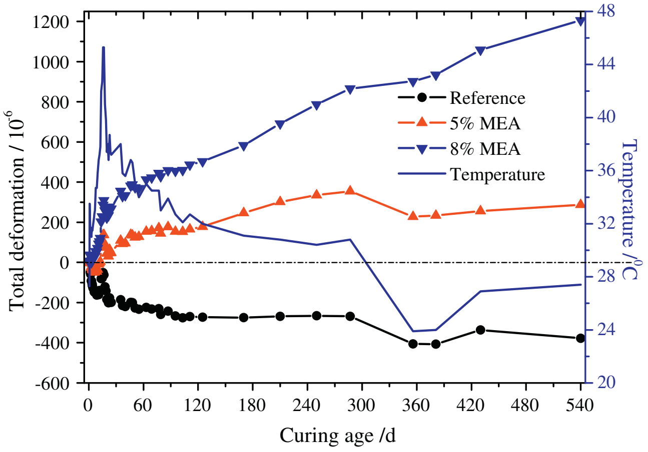
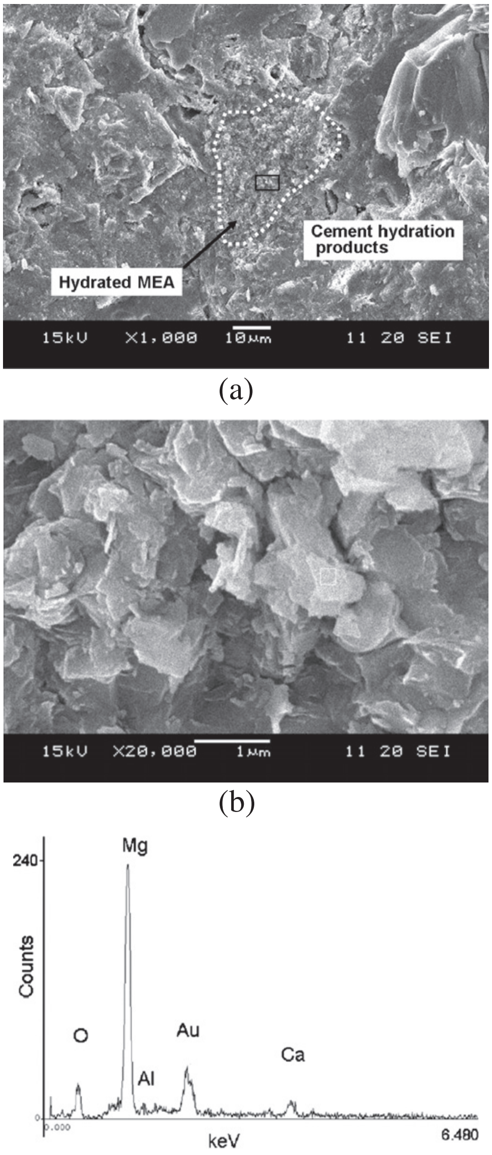


Fig. 7. Total deformations of fly ash cement pastes containing various contents of MEA.

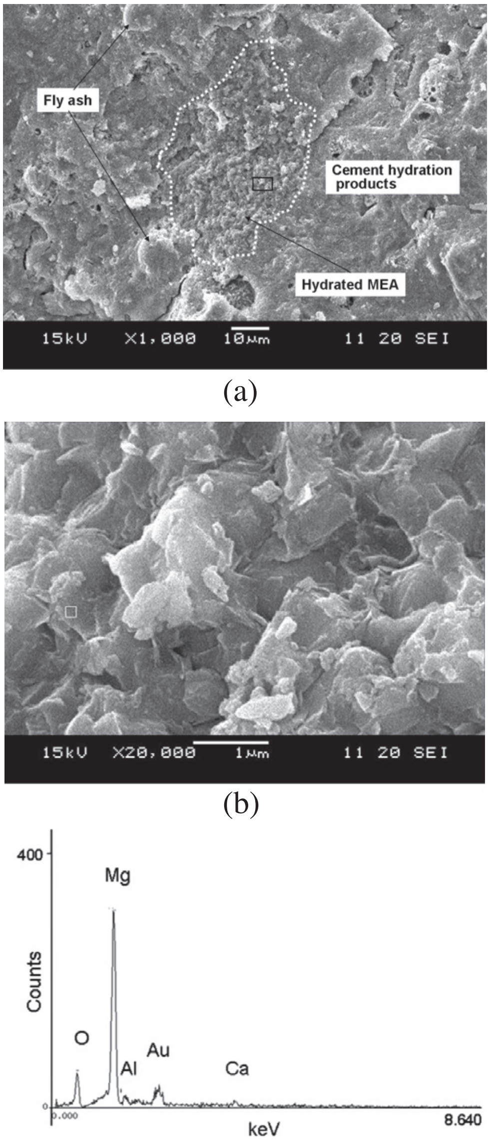


(a)

(b)

(c)

Fig. 8. SEM images of Portland cement paste containing 8% of MEA (a) Hydrated MEA particle embedded in cement hydration products. (b) Higher magnification of the area selected by the black square in (a). (c) EDS analysis of the selected area.



(a)

(b)

(c)

Fig. 9. SEM images of fly ash cement paste containing 8% of MEA (a) Hydrated MEA particle embedded in the cement hydration products. (b) Higher magnification of the area selected by the black square in (a). (c) EDS analysis of the selected area.

went down at the beginning of the cooling stage due to the large thermal shrinkage as a result of sharp temperature declining. Obviously, the expansion of MgO was insufficient to compensate the quickly developed thermal shrinkage completely at the sharp cooling stage. Nevertheless, the total deformation of cement pastes containing MEA was still expansion. At later age, the temperature of cement paste dropped slowly and there was enough time for the hydration of MEA to generate sufficient expansion to compensate the thermal shrinkage, and hence the total deformation curve continued to go up. Table 2 shows the thermal deformations, autogenous deformations, and total deformations of cement pastes during the cooling stage (from the time of peak high temperature to 540 d). As shown in Table 2, during the cooling stage, sufficient expansions were generated in Portland cement pastes containing MEA, and thus compensated the thermal shrinkage completely. Moreover, some gentle expansions were generated at late age, and larger expansion was caused in the cement pastes containing more MEA (Fig. 6). For cement pastes containing AEA, no compensation effects on the thermal shrinkage were observed (Fig. 6).

Fig. 7 shows the total deformations of fly ash cement pastes containing various contents of MEA. The total deformation development of the blended cement pastes had similar trend to that of the Portland cements. The thermal shrinkages of the fly ash cement pastes were also compensated effectively by the MEA, and gentle expansions were produced at late age as well (Fig. 7, Table 2). The compensation of MEA on the long term thermal shrinkage is closely related to the delayed expansion caused by hydration of MgO in MEA. Due to the slow hydration of MgO, the correspondingly produced expansion was delayed and the expansion could be sustained as long time as the hydration of MgO [19,23]. Therefore the MEA is applicable for compensating the long term thermal shrinkage of mass concrete at late age.

3.3. Hydration and expansion of MEA in cement pastes

Dissimilar to the traditional ettringite-bearing additive, MEA could produce expansion even under the non-wet curing condition.

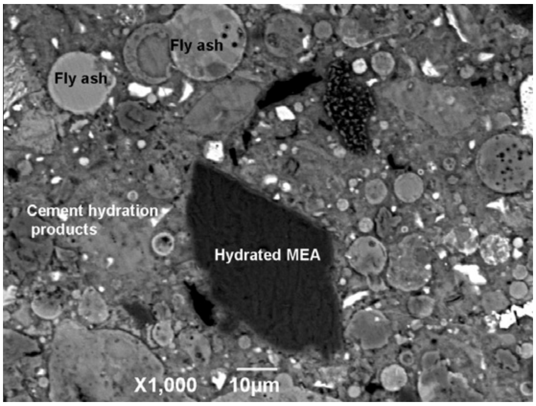


Fig. 10. BSEM image of fly ash cement paste containing 8% of MEA.

This is attributed to that less water is needed for the transformation of MgO into $Mg(OH)_2$ which is the main hydration product of MEA causing expansion. As shown in Eq. (4), the theoretical water-to-MgO ratio for the complete hydration of MgO is 0.45, being less than 0.85 of AEA and closer to the water-to-cement ratio in modern concrete. This indicates that MEA may be more applicable in concrete which is lack of exterior wet curing or has low permeability.

$$MgO + H_2O \rightarrow Mg(OH)_2 \qquad (4)$$

Weight: 40 18.

Water/MgO ratio: 18/40 = 0.45.

Fig. 8 shows the SEM image of Portland cement paste containing 8% of MEA. The hydrated MEA particle was embedded in the cement hydration products (Fig. 8a). As shown in the image at higher magnification, it is obvious that lots of sheet $Mg(OH)_2$ were formed and agglomerated in the hydrated MEA particle (Fig. 8b). Similar to the Portland cement paste, the hydrated MEA particle was also surrounded by the cement hydration products (Fig. 9a), and the sheet $Mg(OH)_2$ was formed and densely agglomerated (Fig. 9b). Fig. 10 shows the BSEM image of fly ash cement paste. Obviously, the hydrated MEA particle was embedded in the cement hydration products. Accordingly, it is conceivable that the MEA hydrated locally, and thus the hydration products of MEA were mainly located in the space that originally occupied by the MEA particle. Therefore the self-expansion of MEA was caused by the transformation of MgO into $Mg(OH)_2$, and moreover the expansion was locally restrained by the surrounding cement matrix, consequently inducing the expansive stress and expanding the cement matrix.

4. Conclusions

Effects of MEA on compensating the autogenous shrinkage and thermal shrinkage of cement pastes were investigated, in which the samples were cured under the non-wet curing condition provided by sealing the samples and preventing the moisture exchange with the environment. Moreover, the microstructures of cement pastes containing MEA were studied by SEM and BSEM. The following conclusions were drawn:

1. Under the non-wet curing condition, both the autogenous shrinkage and long-term thermal shrinkage of Portland cement paste with low water-to-cement ratio were compensated effectively by the expansion generated by the MEA produced in the present work, which was attributed to the relatively low water requirement for the hydration of MgO and its slow hydration rate. The autogenous shrinkage and thermal shrinkage of fly ash cement paste were compensated effectively by the MEA as well, regardless of the incorporation of fly ash. Expansions were generated at late age in all the cement pastes containing MEA, and greater additions of MEA in the cement paste caused higher expansions.
2. Hydration of a MEA particle surrounded by the cement hydration products may cause self-expansion of MEA particle due to the transformation of MgO into $Mg(OH)_2$. The expansion is locally restrained by the cement matrix, and thus resulting in expansive stress and causing macro-expansion of the cement paste.

This study only regards cement paste. However, to facilitate the application of MEA in concrete, further studies on the expansion and mechanical properties (i.e. the compressive stress–strain relationship and tensile stress–strain relationship) of concrete containing MEA under restraint conditions posed by steel reinforcing bars are very important, some of which are now being performed by our group.

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