

DAMAGE BEHAVIOR OF CALCIUM SILICATE SLAG-BASED ALKALI-ACTIVATED MATERIALS EXPOSED TO ELEVATED TEMPERATURES

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Alkali-activated materials (AAMs) were synthesized from calcium silicate slag (CSS), ground granulated blast furnace slag (GGBFS) and class F fly ash (FA). The damage behavior, including the appearance, compressive strength, phase changes and microstructure evolution, of CSS-based AAMs exposed to pre-heated temperatures up to 1000°C were investigated. The findings showed that the microstructure and the compressive strength gradually degraded with elevated temperature from 20°C to 800°C, while upgraded during 800°C to 1000°C. The cracks induced by the quick dehydration of f-H₂O were the main reasons for the strength deterioration as the exposed temperature was lower than 600°C. The continuous dehydroxylation of gel products, incompatibility between shrinking gel products and expanding quartz, as well as the decomposition of calcite resulted into the severe microstructure and strength deteriorations from 600°C to 800°C. As the exposed temperature was elevated from 800°C to 1000°C, the phase transition of gel products into crystal phases e.g. β-wollastonite (CaSiO₃) and gehlenite (Ca₂Al₂SiO₇) contributed to the further deterioration of CSS-based AAMs. Furthermore, the molten phases filled into cracks and bonded the particles together. This contributed to the increasing of density and compressive strength of AAMs matrix.

Keywords: alkali-activated materials; calcium silicate slag; elevated temperatures; damage behavior; microstructure

1. Introduction

Calcium silicate slag (CSS), mainly chemically composed of CaO and SiO₂, and mineralogically composed of 2CaO·SiO₂ (C₂S), is the solid waste of the alumina extraction from high alumina fly ash project which is based on combined pre-desilication and lime-soda sintering process [1]. Therefore, CSS is considered as a potential raw material for building materials [2]. Considering the unneglectable alkali residues (approximately 4 wt%) due to the technology process [1], to synthesize alkali-activated materials (AAMs) with CSS might be an effective approach for its further utilization [3]. The CSS-based AAMs presented excellent properties and great application potentiality in marine environments [4, 5], however, the performances of CSS-based AAMs, such as resistance to higher temperature and corrosive environments, are still unexplored.

Fire damage of structures is one of the most critical threatens to the people and property. Recently, the fire resistance of buildings (especially the high-rise buildings) has drawn more and more attentions with the rapid urbanization [6]. Cement is the basic binder material of concrete and buildings. The resistance of cement to elevated temperatures is one of the key factors determining the fire resistance of buildings.

Generally, AAMs are regarded to have better thermal resistance comparing with the traditional Portland cement (PC) [7]. The absence of Ca(OH)₂ is recognized as one of the major reasons for the better thermal resistance of AAMs [8]. Besides, the

low chemical bonding water content and the incredible structure resilience of the reaction products also contribute to the excellent thermal resistance [9, 10].

In fact, the resistance to high temperature of AAMs is dependent on a series of factors like raw material [11-13], alkaline activator [14, 15] and aggregate type [16]. Based on the calcium content in the system, AAMs can be broadly classified into high-calcium (Si + Ca) system and low-calcium (Si + Al) system. The former mainly produces linear calcium silicate hydrates (C-S-H gels), and the latter mainly yields three-dimensional zeolite-like alkaline aluminosilicate hydrates (N-A-S-H gels) [17, 18]. In recent years, more and more solid wastes, like metallurgical industrial wastes [13, 19] and municipal wastes [13, 20], were successfully utilized in AAMs preparation. The AAMs can be classified as the high-calcium (Si + Ca) and low-calcium (Si + Al) composite system in most cases [12, 13, 21]. Accordingly, the reaction products become the mix of C-S-H gels and N-A-S-H gels known as (N, C)-A-S-H gels [10, 22], which generally provides significant benefits over the N-A-S-H gels alone [9]. Besides, the other components in raw materials (magnesium, aluminium and ferrum, etc.) also play important roles in the volume stability and fire resistance of AAMs [19, 23-25]. Therefore, the resistance of AAMs to high temperatures should be analyzed based on the specific circumstance.

In this study, the damage behavior of CSS-based AAMs exposed to elevated temperatures was studied. The specimen appearance,

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Table 1

Chemical composition of raw materials / wt%										
Materials	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	LOI*	Σ
CSS	25.29	2.55	7.44	48.25	2.39	0.34	3.30	0.55	8.80	98.91
GGBFS	33.50	1.10	12.52	37.90	9.29	-	-	2.51		96.82
FA	42.85	4.32	41.26	3.89	0.56	-	-	0.16	1.25	94.29

* - Loss on ignition

compressive strength, phase changes and microstructure developments of CSS-based AAMs with the elevating of temperature were experimentally investigated. The findings of this study will greatly contribute to the further application of CSS-based AAMs in high temperature environment.

2. Experimental materials and methods

2.1. Materials

Original CSS was supplied by Inner Mongolia Datang International Recycling Resource Development Co., Ltd, China. Ground granulated blast furnace slag (GGBFS) was purchased from China Baowu Steel Group Corporation Limited. Class F fly ash (FA) was obtained from Henan Xing'an New Building Materials Co., Ltd, China. The chemical composition of CSS, GGBFS and FA is shown in Table 1.

The XRD patterns of raw materials are presented in Fig. 1. CSS is mainly consist of larnite (β -C₂S), calcite (CaCO₃) and katoite (Ca₃Al₂(SiO₄)(OH)₈, C₃ASH₄). GGBFS is mostly composed of vitreous phase and FA is mainly consist of mullite (3Al₂O₃•2SiO₂), quartz (SiO₂) and amorphous glass.

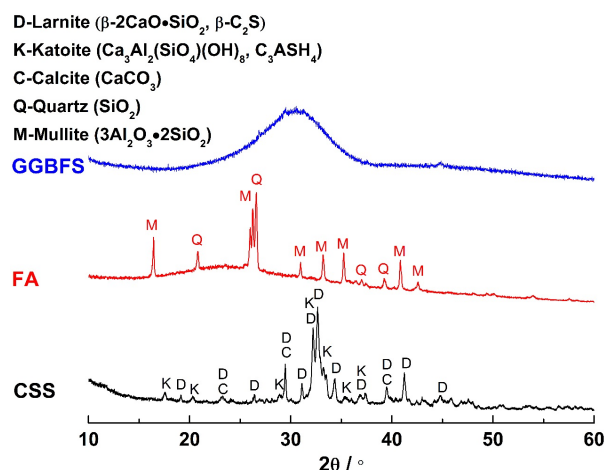


Fig. 1 - XRD patterns of raw materials

Original CSS was dried and then grounded in a laboratory ball mill to obtain the CSS powder. The particle size distributions of CSS, GGBFS and FA are given in Fig. 2.

Technical grade liquid sodium silicate purchased from Beijing red star chemical building materials Co., Ltd. was used as the alkaline activator. The Ms (molar ratio of SiO₂ to Na₂O) is 2.40 and the solid content is 48.97 wt%. The

sodium silicate solution with a Na₂O/H₂O mass ratio of 0.1 was prepared 24 h before using by adding fresh water into the stirring liquid sodium silicate.

The ISO quartz sand from Xiamen ISO Standard Sand Co., Ltd. was used as the fine aggregate in mortars.

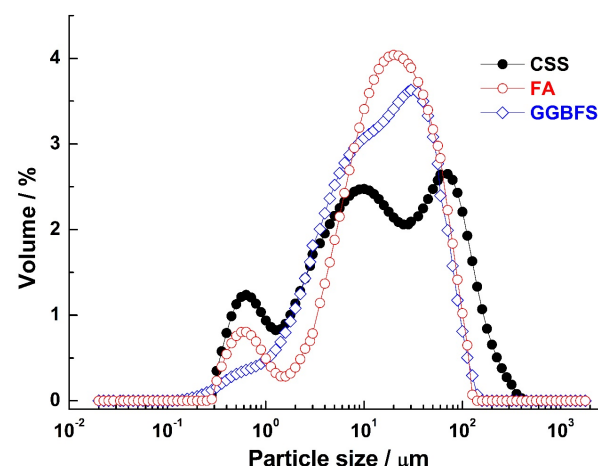


Fig. 2 - Particle size distributions of raw materials.

2.2. Specimen preparation

The CSS-GGBFS-FA blend was premixed with a CSS: GGBFS: FA mass ratio of 52.5: 22.5: 25.0 according to previous research [5], then the CSS-based alkali-activated materials were synthesized by mixing the blend and the sodium silicate solution at a constant water-to-blend ratio (W/B) of 0.5. The pastes sizing 40 mm × 40 mm × 40 mm and mortars sizing 40 mm × 40 mm × 160 mm were prepared in accordance with the Chinese standard GB/T 1346-2011 [26] and GB/T 17671-1999 [27], respectively. The paste and mortar specimens were immediately transferred into a curing cabinet at 20 ± 1°C, RH ≥ 95%. After 24 h, the specimens were demolded and transferred back to the curing cabinet. The specimens were taken out from the cabinet at 21 days and stored in the ambient environment (T = 20 ± 1°C) until 28 days.

2.3. Test methods

In order to evaluate the damage behavior of CSS-based AAMs exposed to elevated temperatures, the specimens were exposed to 20°C (Control), 200°C, 400°C, 600°C, 800°C and 1000°C for 2.5 h, respectively. The exposure was achieved by direct placement in a muffle furnace pre-heated to the test temperature. The specimens were cooled in air to ambient temperature after removing from the muffle furnace. Then the paste

specimens were crushed and ground with an agate mortar for the further phase analysis, while the mortar specimens were used for the surface and section appearances, compressive strength and microstructure analyses.

The compressive strength of mortar specimens was performed with a TYE-300D anti-folding and compression testing machine (Six replicates per data). The X-ray diffraction patterns were collected with a D8 automated diffractometer of Bruker AXS (Cu-K α radiation). The tests were run in the range of 10 - 60° at a step size of 0.02° and a scanning rate of 8 °/min (2 θ). The comprehensive thermal analyses (TG/DSC) were carried out with NETZSCH STA 449C from 30°C to 1000°C at a heating rate of 10 °C/min in N₂. The microstructure analyses were conducted on FEI Quanta 250 FEG Scanning Electron Microscope (SEM) at 20 kV.

3. Results and discussion

3.1. Surface and section appearances

Mortar specimens' morphologies of surfaces and sections are presented in Fig. 3. There were no evident changes in the surface appearance of mortar specimens exposed to temperatures lower than 600°C. However, distinct network-like cracks were observed on the surface as the exposed temperature was higher than 600°C.

Regular changes were observed in the cross section of mortar specimens with the elevating of exposed temperature. The quartz sands were closely bonded together by the binder at 20°C, resulting into a compact hardened matrix. Due to the spalling of the binder, the brightness of quartz sands increased with the exposed temperature from 20°C to 800°C, which indicates the gradual degradation of bonding behaviors between the binder and quartz sands. Nevertheless, it seemed that the separated quartz sands were rebonded together by the binder as the exposed temperature was further elevated to 1000°C. This might be

attributed to the melting of the binder at high temperature. These regular results provided evidences for the bond behavior evolution of mortar specimens as exposed to elevated temperatures.

3.2. Compressive strength

The residual compressive strength and the strength retention ratio of mortar specimens are shown in Fig. 4.

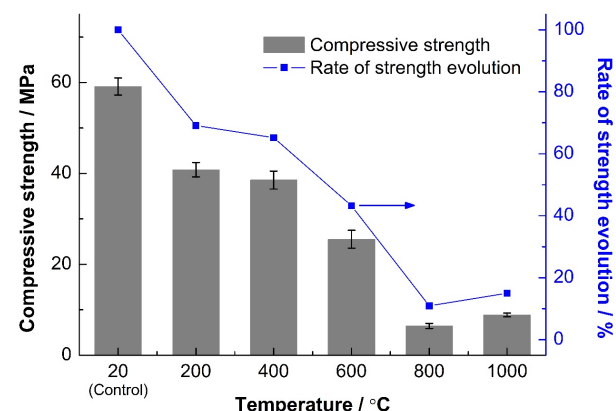


Fig. 4 - Compressive strength of CSS-based alkali-activated mortars exposed to elevated temperatures.

The compressive strength of CSS-based AAMs exposed to elevated temperatures gradually deteriorated from 20°C to 800°C, while slightly improved from 800°C to 1000°C. Besides, the strength retention ratio varied significantly with the exposed temperature. The compressive strength of mortar specimens exposed to 20°C (Control) reached about 60.0 MPa. A significant strength decrease by about 30% was detected as the mortar specimens were exposed to 200°C. As the exposed temperature was elevated to 400°C, only a slight strength decrease was observed. When the exposed temperature was further elevated to 600°C and 800°C, the compressive strength of

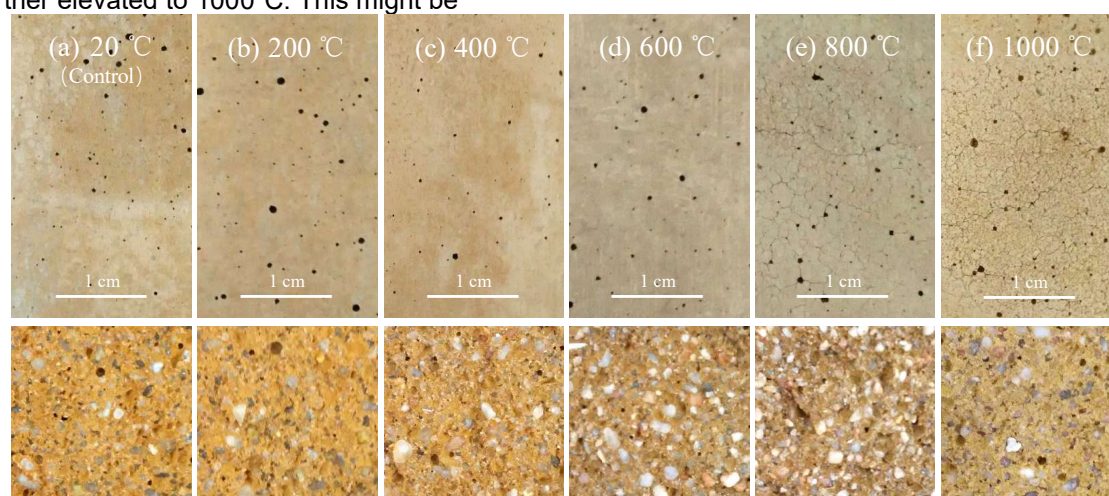


Fig. 3 - Surface and section appearances of mortars exposed to elevated temperatures

mortar specimens remarkably decreased to 40% and 10% of the control, respectively. However, a slight strength increase by around 10% of the control was detected in the mortar specimens exposed to 1000°C. The results of compressive strength are consistent with the surface appearance results mentioned above, which will be explained with the experimental investigation on the phase changes and microstructure evolution analyses.

3.3. Phase changes

The XRD patterns of CSS-based AAMs exposed to elevated temperatures are presented in Fig. 5. The main mineral composition of CSS-based AAMs (Control) is larnite, calcite, katoite, mullite and quartz, which derived from the raw materials. This implied that CSS-based AAMs mainly produced amorphous reaction products. The unreactive crystalline minerals like mullite and quartz were detected in all the patterns. The mineral composition of specimens barely changed as the exposed temperature elevated from 20°C to 600°C. However, the intensity of characteristic peaks of calcite distinctly declined as the specimens were exposed to 600°C, indicating the partly decomposition of calcite. The characteristic peaks of calcite disappeared as the exposed temperature was elevated to 800°C. Meanwhile, the weak peaks characteristic of metastable carnegieite (NaAlSiO_4) were detected. As the exposed temperature was further elevated to 1000°C, the peaks characteristic of carnegieite disappeared, while the crystalline phases like gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$), β -wollastonite (CaSiO_3) and nepheline (NaAlSiO_4) were detected. It should be noted that the decomposition products of calcite are lime (CaO) and carbon dioxide (CO_2). However, there were no CaO or any other calcium-containing phases detected in the specimens exposed to 800°C besides the phases from raw materials. It was reported that the CaO in AAMs can even be detected after exposing to 1000°C [28]. Thus, the un-detection of CaO derived from the decomposition of calcite in this study might be attributed to the limit amounts and the peaks overlapping in XRD patterns.

The TG/DSC curves of CSS-based AAMs are given in Fig. 6. Three endothermic peaks with continuous weight loss were detected in the temperature range from 30°C to 720°C. Besides, two exothermic peaks without weight loss, one distinctly centered at around 820°C and the other weakly centered at around 930°C, were detected.

The distinct peak at around 130°C is assigned to the evaporation of $f\text{-H}_2\text{O}$ that adsorbed on the surface of grains [16]. It is estimated that the evaporation of $f\text{-H}_2\text{O}$ is not the main cause of the damaging stresses, although the $f\text{-H}_2\text{O}$ provides about 60% of the total water content in AAMs [29, 30]. The strength of fly ash/metakaolin-based AAMs can even be increased due to the strengthening effect caused by the geopolymerisation acceleration during drying at high temperature [11, 21]. However,

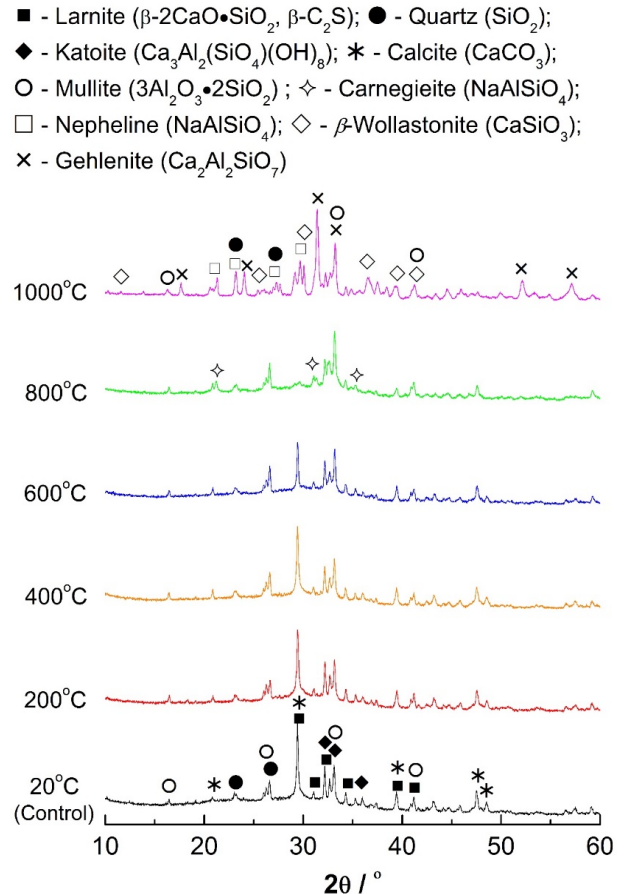


Fig. 5 - XRD patterns of CSS-based AAMs exposed to elevated temperatures.

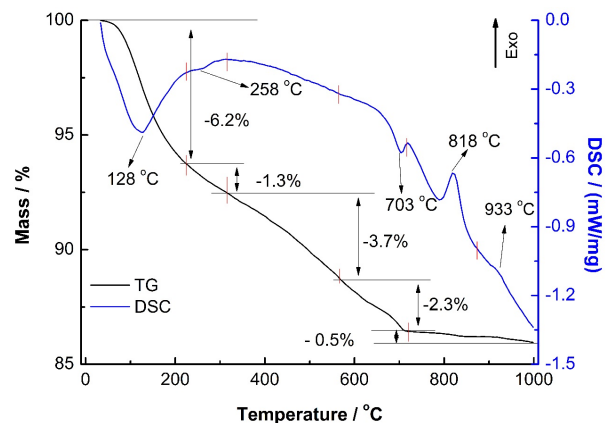


Fig. 6 - TG/DSC curves of CSS-based AAMs

the significant cracking induced by the $f\text{-H}_2\text{O}$ evaporation and the consequent strength losses were also reported in some studies [16, 31]. Because of the low capillary porosity, there even might be a risk of explosive spalling as AAMs are exposed to high temperatures [32]. Therefore, considering the barely changed mineral composition (Fig. 5), the distinct strength deterioration (about 30% of the control, as shown in Fig. 4) can be attributed to the evaporation of $f\text{-H}_2\text{O}$ as the specimens were exposed to 200°C.

The weak peak at about 260°C is associated with the release of chemical bonding H₂O in the products [30]. Accordingly, only a slight strength decrease was detected as the exposed temperature was elevated to 400°C. This might be attributable to the slight shrinkage caused by the dehydration of chemical bonding H₂O.

The peak at approximate 700°C is assigned to the decomposition of calcite into CaO and CO₂. This is confirmed by the XRD results as the exposed temperature was higher than 600°C. The decomposition of calcite would lead to a more porous structure, which could partly contribute to the distinct strength deterioration of CSS-based AAMs as the specimens were exposed to 600°C and 800°C.

The exothermic peaks without weight loss at around 820°C and 930°C are associated with the crystallization of amorphous products. According to previous research [5], CSS-based AAMs mainly produced amorphous C-(A)-S-H gels. Therefore, the exothermic peaks at around 820°C and 930°C are attributed to the crystallization of C-S-H gel into β -wollastonite and C-A-S-H gel into gehlenite, respectively [12, 33]. This is consistent with the XRD results mentioned above. The bonding ability loss caused by this products transition into crystal phases is one of the reasons for the serve strength deterioration. Moreover, it is reported that Ca readily displaces Na by ion exchange as adding calcium into N-A-S-H gels system, and Na will only be retained if insufficient Ca is available [34]. Nevertheless, C-A-S-H gels and N-A-S-H gels can still coexist as (C, N)-A-S-H gels in some specific conditions [12, 22, 34, 35]. The CaO/Na₂O mole ratio of CSS-based AAMs is calculated as 5.7 in this study, which is considerably in excess of the Na accommodated in the N-A-S-H gel structure. Thus, the Na⁺ is supposed to be adsorbed on surface sites in isolate gel porosity and inaccessible to the Ca solution. However, the carnegieite and nepheline were detected in the XRD results as the exposed temperature was higher than 800°C, indicating the presence of N-A-S-H gels in the system [36, 37]. This also partly contributed to the exothermic peaks at around 820°C.

3.4. Microstructure evolution

The SEM micrographs of CSS-based AAMs exposed to elevated temperatures are presented in Fig. 7. The specimens exposed to 20°C exhibited a highly compact microstructure due to the large amount of reaction products. The network-like cracks were observed in the specimens exposed to temperatures lower than 800°C. The number of cracks increased with the exposed temperature from 200°C to 800°C, giving rise to a more and more loose microstructure. The specimens exposed to 800°C even showed a nearly broken microstructure due to the increase of crack amount and width.

The CSS-based AAMs were directly placed into a muffle furnace pre-heated to the test

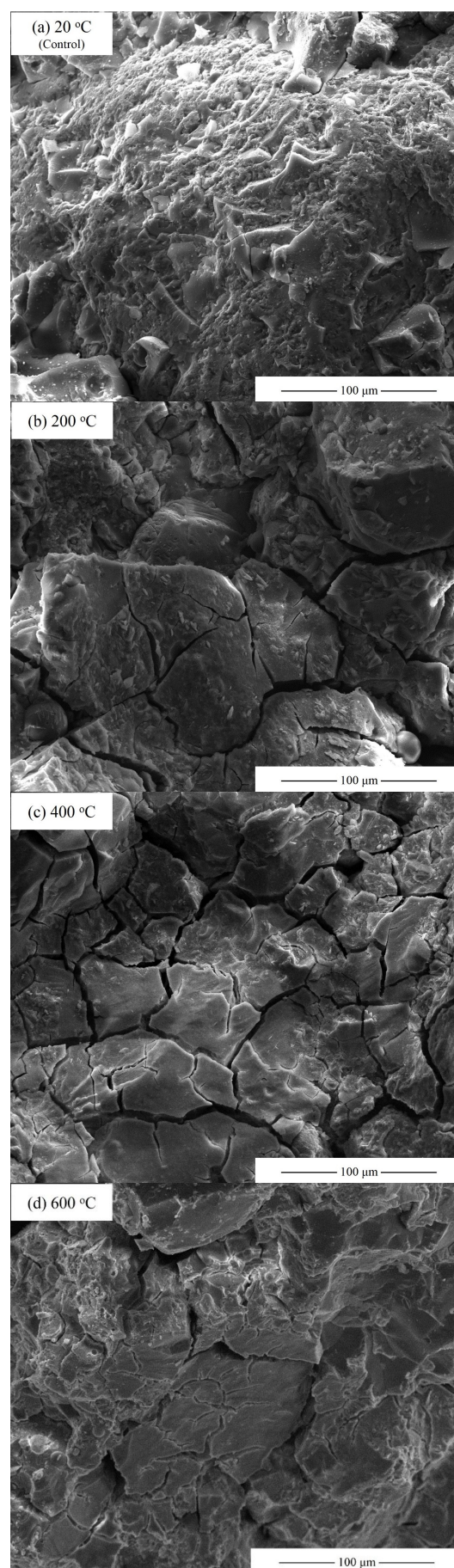


Fig. 7 continues on next page

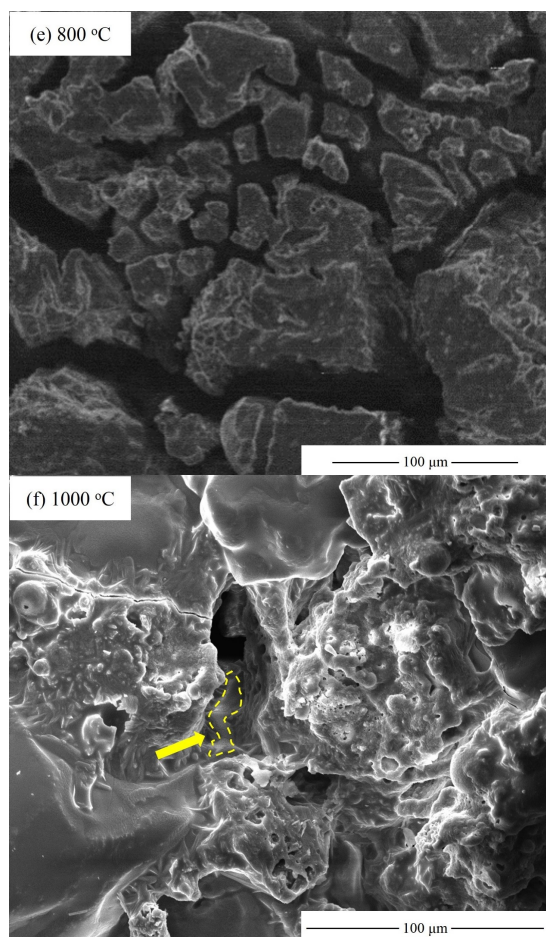


Fig. 7 - SEM micrographs of CSS-based AAMs exposed to elevated temperatures.

temperature in this study. The dramatic differences between ambient temperature and exposed temperatures contributed to the quick evaporation of $f\text{-H}_2\text{O}$, which might result into an instantaneous damaging pore stress in the extremely short time. This is one of the reasons for the increasing cracking and strength deterioration of CSS-based AAMs in this study [23, 38]. Besides, the continuous shrinkage due to physical contraction of AAMs caused by the dehydroxylation of hydroxyl groups in gel products has been widely reported [8, 38]. This might also contribute to the microstructure and strength deterioration. It should be noted that the β -quartz, which mainly derived from aggregates, transforms to α -quartz at 573°C causing a 0.82% thermal expansion [8]. As the mortar specimens were exposed to 600°C and 800°C, the incompatibility between expanding quartz and shrinking gel products was also one of the major reasons for the significant deterioration of microstructure and strength.

The melting phenomenon was observed as the exposed temperature was further elevated to 1000°C. Although the crystallization of C-S-H gels and C-A-S-H gels detected in this temperature range favors the strength deterioration of AAMs, the molten phases filled into the cracks and

bonded the separated solid binders and aggregates together. This increased the compactness of matrix and consequently gave rise to the strength increase of CSS-based AAMs as shown in Fig. 4.

4. Conclusions

The damage behavior of CSS-based AAMs exposed to elevated temperatures from 20°C to 1000°C were studied in this study. Based on the results obtained, the conclusions could be drawn as follows:

(1) The strength of CSS-based AAMs gradually degraded to 10% of the control with the temperature from 20°C to 800°C, while increased by about 10% of the control as the exposed temperature elevated from 800°C to 1000°C.

(2) The cracks derived from the evaporation of $f\text{-H}_2\text{O}$, the expanding quartz sands and the crystallization of gel products resulted into the microstructure and strength deteriorations of CSS-based AAMs as exposed to elevated temperatures from 20°C to 800°C.

(3) The gel products of CSS-based AAMs, mainly C-(A)-S-H gels, transformed into crystal phases like β -wollastonite and gehlenite in the temperature range from 800°C to 1000°C. The molten phases filled into the cracks and bonded the solid particles together which resulted in the matrix density and strength increasing.

Acknowledgement

The authors would like to thank the financial supports from National Natural Science Foundation of China (51572252 and 51402279).

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