

Effect of Al₂O₃, MgO and SiO₂ on Sintering and Hydration Behaviors of CaO Ceramics

Churl Hee Cho, Bong Jin Goo, Kee Sung Lee and Do Kyung Kim[†]

Department of Materials Science and Engineering, Korea Advanced Institute
of Science and Technology, Taejeon 305-701, Korea
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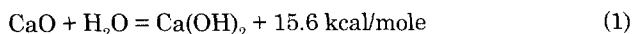
ABSTRACT

CaO ceramics were prepared by conventional sintering process and their hydration behaviors were evaluated by measuring weight increment on saturated water vapor pressure at ambient temperature. CaCO₃ and limestone were used as CaO source materials and Al₂O₃, MgO and SiO₂ were added as sintering agents. Al₂O₃ was a liquid phase sintering agent to increase densification and grain growth rates, whereas MgO and SiO₂ were densification and grain growth inhibitors. Regardless of composition, all of the prepared CaO ceramics showed the improved hydration resistance as bulk density increased. Especially, when bulk density was more than 3.0 g/cm³, there was no weight increment after 120 h of hydration. Therefore, to decrease contact area between CaO and water vapor by increasing bulk density with the Al₂O₃ sintering additive was effective for the improvement of CaO hydration resistance.

Key words : CaO, Conventional sintering, Hydration behavior, Al₂O₃, Bulk density

1. Introduction

Lime (CaO) has been taken much attention as an alkaline refractory that can replace MgO refractory, because it has high melting point (above 2500°C), low oxygen dissociation and vapor pressure, high thermal shock and slag corrosion resistance and high thermodynamic stability in presence of carbon. Even though above-mentioned good properties, its industrial applications were restricted due to its exothermic hydration reaction in contact with water, as represented in equation (1).



The fast CaO hydration reaction originates in its structural instability. CaO has a Face-Centered Cubic (FCC) structure. Ca²⁺ ions are too large to stably position in the 6 coordination sites, so that CaO tends to react with others (H₂O and CO₂) into the more stable forms (Ca(OH)₂ and CaCO₃).

Rice¹⁾ has reported about phenomenological hydration behaviors of CaO ceramics. In porous CaO samples, water vapor penetrated into all parts of samples. On the other hand, in dense CaO samples, isolated white spots were randomly generated on overall sample surface and near the spots, radial cracks nucleated and grew to finally produce a

white powder. After the radial cracks forming, hydration rate sharply increased due to formation of Ca(OH)₂ on crack surface. Cutler *et al.*²⁾ have also reported that CaO hydration reaction was activated by structural destructions due to volumetric expansion. Beruto *et al.*^{3,4)} have reported that CaO powders reacted with water vapor at 25°C to produce Ca(OH)₂ powders from XRD analysis. Ca(OH)₂ powders were formed by solution and precipitation mechanism of initial CaO powders. Also, on considering that the volumetric expansion in CaO hydration reaction occurred in perpendicular direction to crack surface, CaO hydration was studied in the viewpoint of structural change. The close packed plane (111) and its perpendicular direction [111] in FCC CaO structure were compared with the close packed plane (001) and its perpendicular direction [001] in HCP Ca(OH)₂ structure, respectively. In the hydration reaction of CaO to Ca(OH)₂, dimensional mismatch between the close packed planes is very small (3.4 → 3.59 Å). On the other hand, dimensional mismatch between the perpendicular directions is relatively large (2.78 → 4.91 Å). Therefore, there is about two times of volume expansion in the CaO hydration reaction because of a structure expansion of Ca(OH)₂ in the [001] direction. Ewing *et al.*⁵⁾ have investigated about the decomposition of CaCO₃ and Ca(OH)₂ powders to CaO in vacuum at 510 to 650°C and 320°C, respectively. According to the evaluation of specific surface area and porosity, the decomposed powders were porous CaO agglomerates. Therefore, the formed CaO agglomerates were rapidly hydrated by water vapor infiltrated via internal open pores.

Recently, much attention was taken to the processes to improve hydration resistance of CaO, because CaO is

[†]Corresponding author : Do Kyung Kim

E-mail : dkkim@kaist.ac.kr

Tel : +82-42-869-4118 Fax : +82-42-869-3310

expected to be a filter to remove non-metallic inclusions in the molten steel industry. Up to now, three kinds of processes to improve the hydration resistance were developed: one is to form CaCO₃ coating layers,⁶⁻⁸⁾ another is to form protective layers by liquid-phase sintering⁹⁻¹⁶⁾ and the third is to remove pores by sintering.^{1,17,18)} All the three processes have retarded CaO hydration reaction by decreasing contact area between CaO and water vapor. In the present study, various CaO ceramics were prepared by convention sintering process and then their hydration behaviors were investigated. Effect of Al₂O₃, SiO₂ and MgO additives on sintering and hydration behaviors of CaO, origin of high densification rate of limestone and relationship between sintering density and hydration resistance will be elucidated.

2. Experimental Procedure

As a raw material to prepare pure CaO ceramics, CaCO₃ (> 99%, < 1 μm, High Purity Chemicals, Japan) powder was used. As a CaO source material, limestone, abundantly bared in the earth crust, was also applied. XRF results for limestone were summarized in Table 1. From the loss of ignition, limestone contains 95.50 wt% CaCO₃ with 4.5 wt% impurities, which were K₂O, Na₂O, Fe₂O₃, TiO₂, MnO and P₂O₅ and minor impurities. If the minor impurities are ignored, limestone is composed of 96 wt% CaCO₃ and about 4 wt% major impurities. The major impurities were SiO₂, MgO and Al₂O₃ of which contents were 2.22, 1.14 and 0.66 wt%, respectively.

In addition to CaCO₃ and limestone, CaCO₃-Al₂O₃, CaCO₃-SiO₂, CaCO₃-MgO, CaCO₃-Al₂O₃-SiO₂-MgO mixed powders were used to elucidate effect of each additive on sintering and hydration behaviors of CaO and role of each additive on sintering of limestone. The used additives were Al₂O₃ (> 99.99%, 0.3 μm, High Purity Chemicals, Japan), SiO₂ (> 99.9%, 0.8 μm, High Purity Chemicals, Japan) and MgO (> 99%, 0.3 μm, Mallinckrodt Chemicals, U.S.A.) powders. The overall content of additives was added to be 4 wt% in each mixed powder. Specially, in the CaCO₃-Al₂O₃-SiO₂-MgO mixed powder, amounts of Al₂O₃, SiO₂ and MgO were 0.66, 2.22 and 1.14 wt%, respectively. Its chemical composition was the same to that of limestone. The mixed powders were prepared by wet ball mixing process with using ZrO₂ balls and absolute grade ethanol and then drying in vacuum at 50°C.

CaO ceramics were prepared by conventionally sintering green compacts of the starting powders on inert platinum plates at 1300, 1400, 1500 and 1600°C for 2 h. The green compacts had about 2 g/cm³ of bulk density, were discs with 1 cm of diameter and prepared by cold isostatic pressing

with 150 MPa pressure. Bulk density of CaO ceramics was calculated by measuring their weight, diameter and height. Microstructure, crystalline phase and chemical composition were characterized by SEM(515, Philips, Holland), XRD(D/Max-IIIC, Rigaku, Japan) and EDS(Phoenix, Philips, Holland) analysis, respectively. In SEM analysis, CaO ceramics polished with absolute-grade ethanol to prevent from hydration and then their microstructure images were obtained in the back-scattered electron beam mode. Hydration behavior of CaO ceramics was investigated by monitoring weight increment of CaO ceramics in a box under saturated water vapor pressure at ambient temperature. Water vapor pressure is in range of 7.4 to 12.3 kPa at 20 to 30°C¹⁹⁾ and fluctuation of water vapor pressure was ignored in hydration test.

3. Results and Discussion

3.1. Hydration Behavior

Fig. 1 shows XRD patterns of the samples prepared by sintering CaCO₃ powder at 1300°C for 2 h (a) before and (b) after 120 h of hydration test. The sample before the hydration test was a disc-shaped pure CaO ceramic. On the other hand, the sample after the hydration test was a white pow-

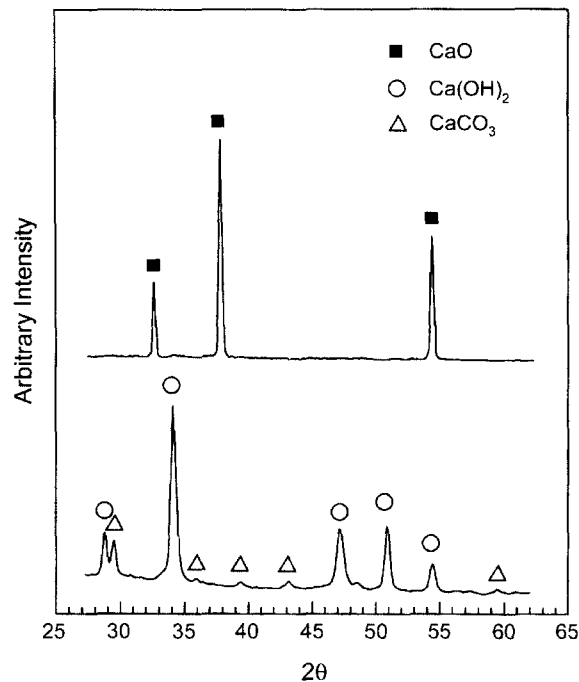


Fig. 1. XRD patterns of CaO samples (a) before and (b) after hydration test.

Table 1. XRF Results of Limestone

Component	CaO	L.O.I.*	SiO ₂	MgO	Al ₂ O ₃	K ₂ O	Na ₂ O	Fe ₂ O ₃	TiO ₂	MnO	P ₂ O ₅	Total
wt%	55.49	38.95	2.29	1.17	0.68	0.17	0.16	0.16	0.02	0.01	0.01	99.11

*Loss of ignition

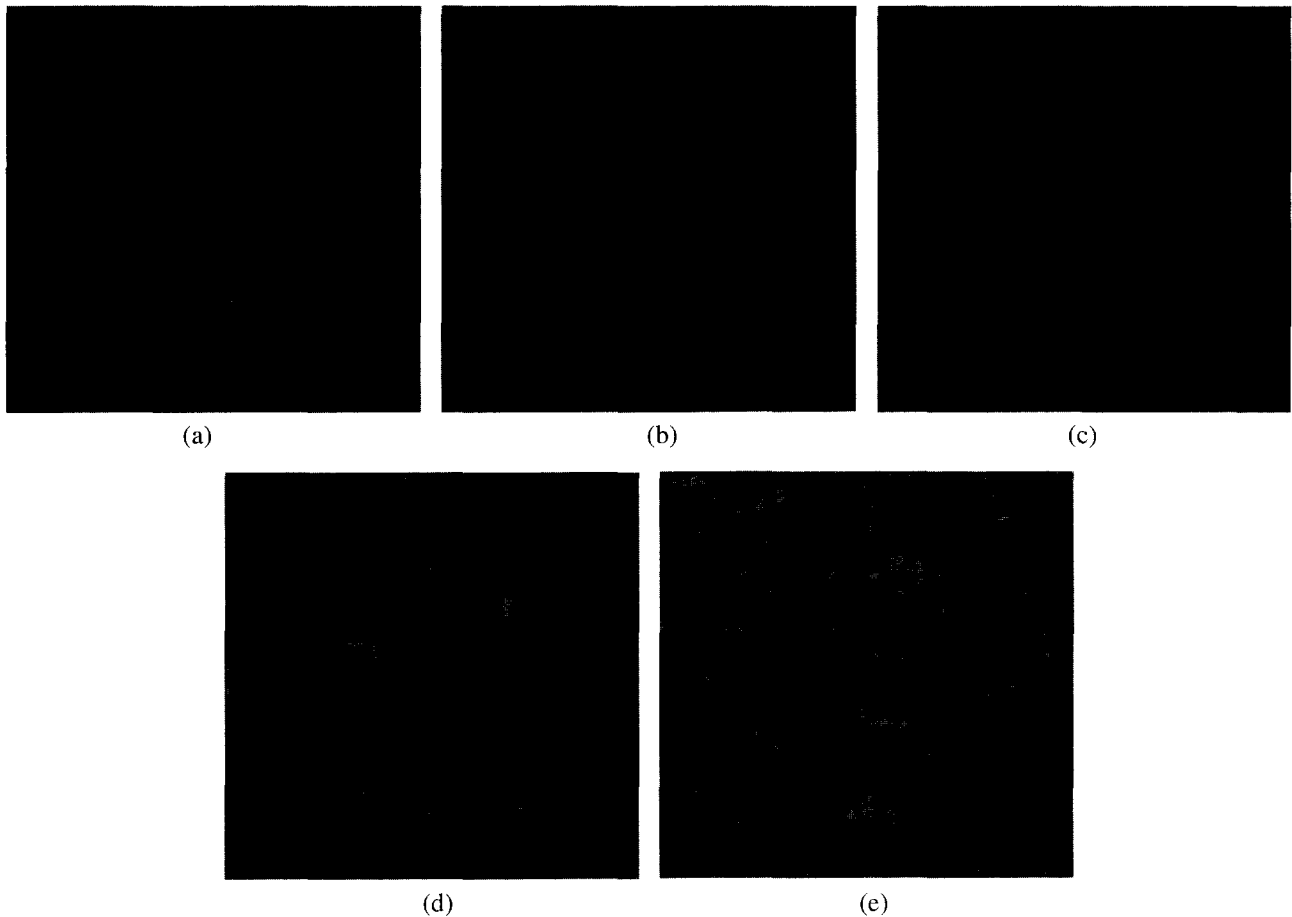


Fig. 2. Photographs of CaO ceramics at (a) initial, (b) spot-shaped surface hydration, (c) local surface cracking and (d) and (e) crushing stages in hydration test.

der and composed of main Ca(OH)_2 and minor CaCO_3 phases. It is remarkable that in 120 h of hydration test, CaO disc was completely destroyed to the powder. Therefore, in the hydration test, CaO reacted with water vapor and CO_2 in air to form Ca(OH)_2 and CaCO_3 phases, respectively.

Fig. 2 shows photographs of CaO ceramics at typical stages in hydration test. Fig. 2(a) is a photograph for the initial stage before hydration. Sample surface was clean and smooth. Fig. 2(b) is a spot-shaped surface hydration stage. In CaO ceramics with low sintering density, white spots already formed on sample surface before hydration test. Especially, spot-shaped surface hydration tends to preferentially occur at circular edges of CaO discs. Fig. 2(c) is a photograph for nucleation and growth stage of local surface cracks. A large circular closed crack tends to form along circular edges due to preferential spot-shaped surface hydration along the edge of CaO discs. But, in some samples, surface cracks randomly formed due to random formation of spot-shaped surface hydration in overall sample surface. Local surface cracks were nucleated and penetrated into interior of samples due to the volumetric expansion of CaO hydration reaction. As shown in Fig. 2(d) and (e), CaO discs

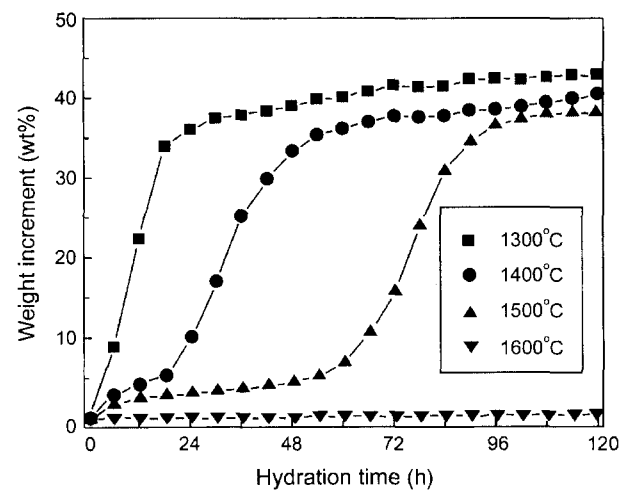


Fig. 3. Weight increment vs. hydration time curves of CaO ceramics in the hydration test; the CaO ceramics were prepared by sintering the CaCO_3 -MgO mixture at 1300, 1400, 1500 and 1600°C for 2 h.

were decomposed into small CaO clusters due to repeated surface hydration and cracking processes. Especially, on the

edge decomposition process occurred due to the preferential circular crack formation. Finally, CaO disc completely hydrated to powder.

Fig. 3 shows weight increment *vs.* hydration time curves of the CaO ceramics prepared by sintering the CaCO₃-MgO mixed powder at 1300, 1400, 1500 and 1600°C for 2 h. As sintering temperature increased, weight gain decreased. In all samples, weight was changed with three regions: one is the initial slow weight increment region, another is the sharp weight increment region and the third is the slow weight saturation region to about 40 wt%. The first slow weight increment regime was from the spot-shaped surface hydration, the second came from the nucleation and growth of surface cracks and crushing and the third was related to the completion of hydration. These results were well accordance with Rice's results.¹¹ In a word, CaO hydration was activated by formation and penetration of surface cracks stemmed from the volumetric expansion of hydration reaction.

3.2. Sintering Behavior

CaO ceramics were prepared by conventional sintering of CaCO₃, limestone, CaCO₃-MgO, CaCO₃-SiO₂, CaCO₃-Al₂O₃, CaCO₃-Al₂O₃-SiO₂-MgO starting powders. Fig. 4 shows bulk density *vs.* sintering temperature curves of CaO ceramics sintered at 1300, 1400, 1500 and 1600°C for 2 h. Limestone, CaCO₃-Al₂O₃ and CaCO₃-Al₂O₃-SiO₂-MgO powders showed fast densification behavior. On the other hand, CaCO₃, CaCO₃-SiO₂ and CaCO₃-MgO powders did not show good densification behavior. Therefore, Al₂O₃, a common impurity in limestone, CaCO₃-Al₂O₃ and CaCO₃-Al₂O₃-SiO₂-MgO powders, was suggested to be a good sintering additive to accelerate CaO densification.

Fig. 5(a) and (b) show SEM photographs of the CaO ceramics prepared by sintering the CaCO₃ and limestone powders at 1600°C for 2 h, respectively. As shown in Fig.

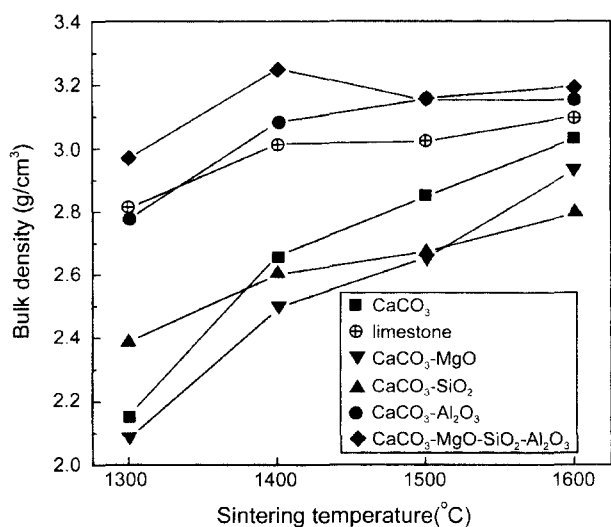


Fig. 4. Bulk density *vs.* sintering temperature curves of the prepared CaO ceramics.

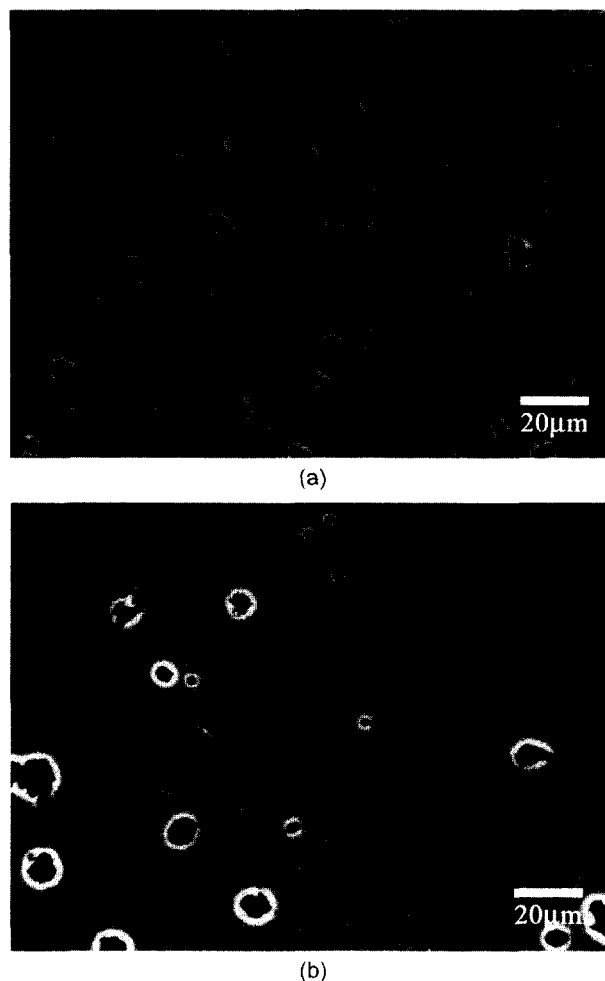
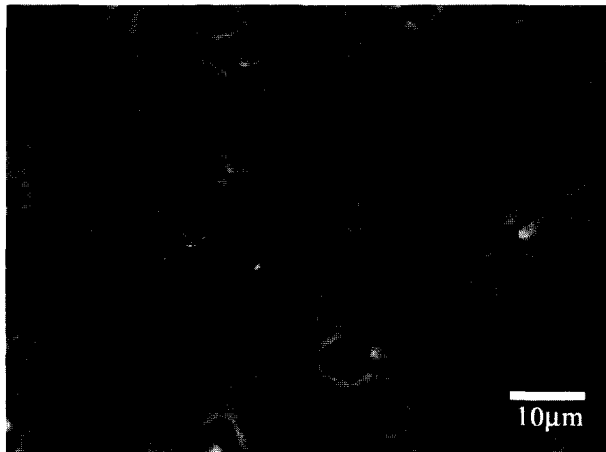


Fig. 5. SEM photographs of the CaO ceramics prepared by sintering the (a) CaCO₃ and (b) limestone powders at 1600°C for 2 h.

5(a), the CaO ceramic prepared from CaCO₃ powder had many rounded closed pores with about 10 mm of diameter. From EDS composition analysis, solid region around the rounded pores was composed of Ca(50.3 at%) and O (49.7 at%) elements. The atomic ratio of Ca to O was nearly 1, so that the solid region was made of CaO phase. Fig. 5(b) is a SEM photograph of the CaO ceramic prepared from the limestone. The CaO ceramic was composed of rounded grains, grain boundary phase and closed pores. The closed pores were about 10 mm sized. The rounded grains were about a few 10 mm sized in diameter and surrounded by the continuous grain boundary phase. The continuous grain boundary phase was a mixture of the gray-colored and black-colored parts. The gray-colored part was wetted well by the black-colored part. In EDS composition analysis, the gray-colored part was composed of Ca(34.4 at%), Si(12.1 at%) and O(51.1 at%) elements, so that the gray-colored part was a calcium silicate phase. On the other hand, the black-colored part was mainly composed of Ca(26.3 at%), Al(15.3 at%) and O(48.2 at%) elements and



(a)

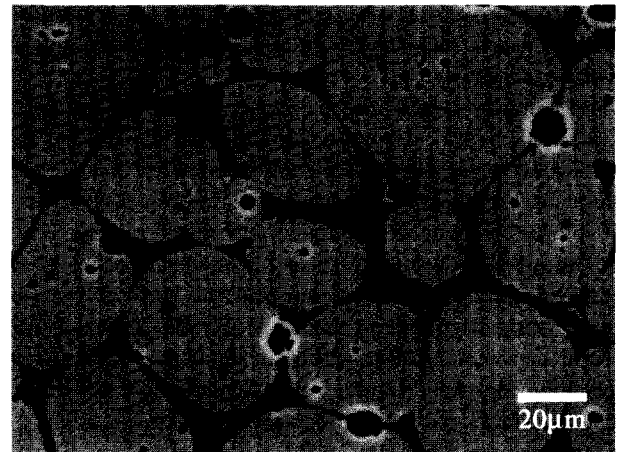


(b)

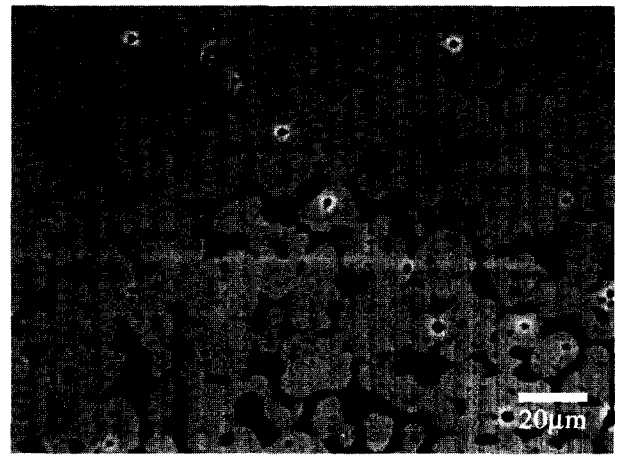
Fig. 6. SEM photographs of the CaO ceramics prepared by sintering the (a) $\text{CaCO}_3\text{-MgO}$ and (b) $\text{CaCO}_3\text{-SiO}_2$ powders at 1600°C for 2 h.

also contained small amounts of minor Mg, Si and Fe elements. On considering that the black-colored part embedded the minor impurities, it was a liquid phase during the sintering process.

Fig. 6(a) and (b) show SEM photographs of the CaO ceramics prepared by sintering the $\text{CaCO}_3\text{-MgO}$ and $\text{CaCO}_3\text{-SiO}_2$ starting powders at 1600°C for 2 h, respectively. In Fig. 6(a), a SEM micrograph of the CaO ceramic prepared from the $\text{CaCO}_3\text{-MgO}$ mixed powder was represented. The microstructure was similar to that of the ceramic prepared from the CaCO_3 powder as shown in Fig. 5(a), besides that it contained more elongated pores. The elongated shape means that the pores are open. From EDS composition analysis, solid region around the pores was composed of Ca(48 at%), O(48.8 at%) and Mg(3.1 at%) elements. The incorporation of Mg in the region means that in the sintering process, MgO formed a solid solution with CaO. Also, compared to the pure CaO as shown in the Fig. 5(a), the low density means that the Mg incorporation into CaO lattice decreases CaO densification rate. Xintian *et*



(a)



(b)

Fig. 7. SEM photographs of the CaO ceramics prepared by sintering the (a) $\text{CaCO}_3\text{-Al}_2\text{O}_3$ and (b) $\text{CaCO}_3\text{-Al}_2\text{O}_3\text{-SiO}_2\text{-MgO}$ powders at 1600°C for 2 h.

*al.*¹¹⁾ have reported similar results about effect of MgO on sintering behavior. In Fig. 6(b), the CaO ceramic prepared from the mixed $\text{CaCO}_3\text{-SiO}_2$ powder was represented. The microstructure was composed of white and gray parts and many open pores. In EDS composition analysis, the white part was composed of Ca(51.1 at%) and O(48.9 at%) elements and the gray part was composed of Ca(39.4 at%), Si(10.3 at%) and O(50.1 at%) elements. Nagayama *et al.*¹²⁾ have reported that the gray part was a calcium silicate phase and successfully inhibited densification and grain growth rates in CaO sintering process. Therefore, the $\text{CaCO}_3\text{-SiO}_2$ powder showed low densification behavior due to formation of the calcium silicate phase.

Fig. 7(a) and (b) show SEM photographs of the CaO ceramics prepared by sintering the $\text{CaCO}_3\text{-Al}_2\text{O}_3$ and $\text{CaCO}_3\text{-Al}_2\text{O}_3\text{-SiO}_2\text{-MgO}$ starting powders at 1600°C for 2 h, respectively. Fig. 7(a) showed a SEM micrograph of the CaO ceramic prepared by sintering the CaCO_3 and Al_2O_3 powder mix at 1600°C for 2 h. The microstructure was similar to that in Fig. 5(b) and composed of 4 to 50 mm sized white

grains and continuous black grain boundary phase. The grain boundary phase completely wetted the rounded white grains. There were micron-sized closed pores in the white grains and about 10 mm sized closed pores appeared in between the black grain boundary and the white grains. In EDS composition analysis, the white grain was composed of Ca(41.7 at%) and O(58.3 at%) element and the continuous grain boundary phase was composed of Ca(32.2 at%), Al(23.6 at%) and O(44.2 at%) elements. Because the sintering temperature, 1600°C, was higher than the eutectic temperature between CaO and Al₂O₃, the continuous grain boundary phase was a liquid phase and played the role of materials migration path in the CaO sintering. Therefore, the Al₂O₃ was acted as a liquid-phase sintering additive, so that the prepared CaO ceramic was much more dense than the pure CaO ceramic. In Fig. 7(b), a SEM photograph of the CaO ceramic prepared from the CaCO₃-Al₂O₃-SiO₂-MgO mixed powder was shown. The microstructure was similar to that of the CaO ceramics prepared from the limestone and the CaCO₃-Al₂O₃ powder and was composed of about 20 mm sized grains, grain boundary phase and a few small closed pores. As like to the CaO ceramic prepared from the limestone, the grain boundary phase was composed of gray-colored grains and a continuous black-colored phase. The continuous black phase completely wetted both the white CaO grains and the gray-colored grains in the grain boundary region. In EDS composition analysis, the gray-colored grain was composed of Ca(39.9 at%), Si(9.1 at%) and O(55.2 at%) elements. And the continuous black phase was composed of Ca(28.8 at%), Al(15.1 at%), O(51.2 at%) and small amounts of Mg and Si elements. The gray-colored grain was calcium silicate phase and the continuous black phase was eutectic melt. These results were same to the CaO ceramics prepared by the limestone, except that the white CaO grains were much smaller. Therefore, in the sintering of the mixed CaCO₃-Al₂O₃-SiO₂-MgO powder, its high densification rate was from the Al₂O₃ component and its slow grain growth rate was from the MgO and SiO₂ components.

Table 2. EDS Results of Each Appeared Phase in the Prepared CaO Ceramics

Sample	Color	Composition (at%)					
		Ca	Mg	Al	Si	Fe	O
CaCO ₃	White	50.3	-	-	-	-	49.7
Limestone	Gray	34.4	-	-	12.1	-	51.1
	Black	26.3	δ	15.3	δ	δ	48.2
CaCO ₃ -MgO	White	48.0	3.1	-	-	-	48.8
CaCO ₃ -SiO ₂	White	51.1	-	-	-	-	48.9
	Gray	39.4	-	-	10.3	-	50.1
CaCO ₃ -Al ₂ O ₃	White	41.7	-	-	-	-	58.3
	Black	32.2	-	23.6	-	-	44.2
CaCO ₃ -MgO-SiO ₂ -Al ₂ O ₃	Gray	39.9	-	-	9.1	-	55.2
	Black	28.8	δ	15.1	δ	-	51.2

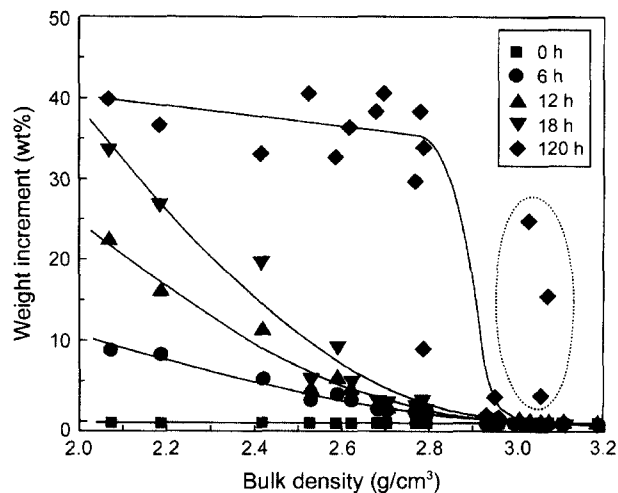


Fig. 8. Weight increment vs. bulk density curves of the prepared CaO ceramics at various hydration times.



Fig. 9. SEM photograph of the CaO ceramic prepared by sintering the CaCO₃-Al₂O₃ powder at 1600°C for 2 h.

In Table 2, the EDS chemical compositions of each appeared phases in each CaO ceramic prepared in the present study were summarized. It is clear that the white-colored, gray-colored and black-colored parts were phases of CaO, calcium silicate and Ca-Al-O melt, respectively.

From the above-mentioned results, it can be concluded that in CaO sintering process, Al₂O₃ is a liquid phase sintering additive to accelerate densification and grain growth rates and MgO and SiO₂ are inhibitors of densification and grain growth due to the formation of solid-solution and calcium silicate, respectively. The formation of solid solution and calcium silicate inhibits materials transfer in CaO sintering.

3.3. Relation between Bulk Density and Hydration Resistance

Fig. 8 shows weight increment vs. bulk density curves of the prepared CaO ceramics at various hydration times. As bulk density increased, weight increment decreased. Especially, all the CaO ceramics with more than 3.0 g/cm³ of

bulk density showed no weight increment after 160 h of hydration test. However, as shown in the dashed ellipse in Fig. 8, the CaO ceramics prepared by sintering the CaCO₃-Al₂O₃ mixed powder at 1400, 1500 and 1600°C for 2 h showed low hydration resistances, even though their bulk densities were above 3.0 g/cm³. A clue for the low hydration resistance of the CaO ceramics prepared from the CaCO₃-Al₂O₃ powder could be found in their microstructure.

Fig. 9 shows a SEM micrograph of the CaO ceramic prepared by sintering the CaCO₃-Al₂O₃ powder at 1600°C for 2 h before hydration test. There was a well developed long crack that was originated in sample surface and branched into sample interior. It was remarkable that the crack was initiated from the regions where Ca-Al-O liquid phase was non-uniformly concentrated on. Therefore, the low hydration resistance of the CaO ceramics prepared by sintering the CaCO₃-Al₂O₃ powder was stemmed from the crack formation and propagation by thermal expansion mismatch between CaO grain and Ca-Al-O grain boundary phase, because the cracks played the role of penetration paths of water vapor to activate CaO hydration.

4. Conclusion

CaO ceramics were hydrated via spot-shaped surface hydration, nucleation and growth of local surface cracks and crushing stages to be powders. In CaO sintering process, Al₂O₃ is a liquid phase sintering additive to accelerate densification and grain growth rates. On the other hand, MgO and SiO₂ are inhibitors of densification and grain growth due to the formation of solid-solution and calcium silicate, respectively. Regardless of composition, all of the prepared CaO ceramics improved their hydration resistance with their bulk density increasing. Especially, when bulk density was more than 3.0 g/cm³, CaO ceramics showed high hydration resistance. Therefore, Al₂O₃ effectively improved sintering behavior and hydration resistance of CaO ceramics.

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