PREPARATIONOF HIGH POROSITY SLUMPING MOLD FROM REFRACTORY MORTAR

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Abstract

Sagging molds or slumping molds are used for putting glass plate onto a third dimension and are used for the production of glassware containers and many kinds of sagging glass articles. The use of slumping molds has been favored by many sagging glass factories in Thailand. However, such molds must be imported athigh cost from abroad. Refractory mortar is a candidate material for making slumping molds due to the easeoffabricationin every shape and size. In this study, dolomite clay, calcinedalumina, and white Portland cement were mixed with distilled water 40 wt% into apaste. The flow ability of the paste was adjusted by the addition of a super plasticizer. The paste waspoured intoplastic containersused as molds and cured at room temperature for 48 h. After being dried in air for 24 h, specimens were dried in an oven at 110°C for 24 h and then were fired at 1000-1250°C in an electrical box furnace. The results show that the proper ratio of raw materials is dolomite clay 40 wt%, calcinedalumina 35 wt%, white portland cement 25 wt%, and water 40 wt%. High porosity specimens with suitable density are achieved when fired at 1250°C. The effects of the firing of the samples on theirchemical property, thermal property, and microstructure will be discussed.

Keywords: Slumping mold, refractory mortar, dolomite clay, alumina, porous

Introduction

Sagging molds or slumping molds are used for puttingglass plate onto a third dimension and are used for the production of glassware containers and many kinds of sagging glass articles. The use of slumping molds has been favored by many sagging glass factories in Thailand. However, such molds must be imported athigh cost from abroad. Refractory

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mortar with low bulk density is a candidate material for making slumping molds due to the ease offabricationin every shape and size.The sagging molds areused at a high temperature of approximately 800°C (Chen and Yi, 2011). They should have thermal stabilityat such a high temperature, a low thermal expansion coefficient, a high enough mechanical strength to thickness ratio, and an easeformachining and smoothing of their surfaces (Wendell, 1977; Chen and Yi, 2011).

In the CaO-Al_2O_3-SiO_2 phase equilibrium diagram (Cheng *et al.*, 2012) consisting of anorthite, gehlenite, mullite, wollastonite, and corundum,anorthite ($CaO.Al_2O_3.2SiO_2$) is anappropriate phase because of its high thermal shockresistance, low thermal expansion coefficient ($4.82\times10^{-6\circ}C^{-1}$), and high flexural strength (Mergen and Aslanoğlu, 2003; Cheng *et al.*, 2012). Anorthitewas investigated from different CaO sources ($Ca(OH)_2$, marble powder, calcite ($CaCO_3$), and gypsum) (Kurama and Ozel, 2009). Ananorthite refractory insulating brick was produced from clay, recycled paper waste, and sawdust by firing at 1200°C. Pores in the brick were created from the burning of cellulose fibers and the decomposition of calcium carbonate (Sutcu *et al.*, 2012).

In this study, high porosity slumping molds have been developed by mixing dolomite clay, calcined alumina, and white portland cement. Dolomite clay and white portland cement are used as the source of CaO and SiO_2 to produce anorthite. Moreover, white portland cement is used for easein casting (mortar casting). Porosity in the body is formed from the decomposition of dolomite and calcium hydroxide (from cement-water).

Materials and Methods

Dolomite clay (Yorin Trading LP, Saraburi, Thailand), calcined alumina (DN-430, Albemarle Corp., Baton Rouge, LA, USA), and white portlandcement (White Tiger, The Siam White Cement Co. Ltd., Bangkok, Thailand) areused as rawmaterials. Table 1 shows the chemical compositions of the raw materials used in this study. The compositions of the samples areshownin Table 2. All of the formulascontained the same amount of 25 wt% white portland cement, 1 wt% superplasticizer (Melflux 2651F, BASF Construction Polymers GmbH, Trostberg, Germany), and 40 wt% distilled water. Figure 1 shows the positions of the compositions FC1, FC2, FC3, FC4, and FC5 in the CaO-Al_2O_3-SiO_2 phase equilibrium diagram (Sutcu and Akkurt, 2010).

Alumina powder, dolomite clay, and the superplasticizerwere mechanicallystirred

Table 1. Chemical compositions of raw materials

Oxide	Calcined alumina (wt%)	Dolomite clay (wt%)	White portland cement (wt%)
SiO_2	0.01	56.80	11.26
Al_2O_3	99.30	13.19	1.77
CaO	0.06	9.87	78.69
MgO	-	3.82	-
Na_2O	0.50	1.32	-
K_2O	-	1.50	0.14
Fe_2O_3	0.02	0.82	0.25
LOI	0.20	12.68	6.14

withdistilled water for 15 min in a plastic container. The well mixed slurry was sieved through #100 mesh and was aged for 24 h. White portland cement was added into the slurry and was stirred for 15 min in a vacuum mixer. Mortar was then cast into plastic molds and was leftfor setting for 24 h. After the setting, specimens were removed from the plastic molds and were leftto dryatroom temperature for 24 h. Specimens were then dried in an oven at 110°C for 24 h and were weighed before firing at 1000-1250°C for 1 h with theheating rate of 10°C/min.

Shrinkages were determined by measuring the length of specimens before drying and after firing.Bulk density, water absorption, and apparent porosity after firing were determined by the Archimedes' method.

The firing behavior of cut 25×5×5 mm samples of all compositions fired in the temperature range of 30-1250°C with a heating rate of 10°C/min in air atmosphere

Table 2. Compositions of refractory mortar samples

Composition (wt%)	Calcined alumina	Dolomite clay	White portland cement
FC1	25	50	25
FC2	30	45	25
FC3	35	40	25
FC4	40	35	25
FC5	35	40	25

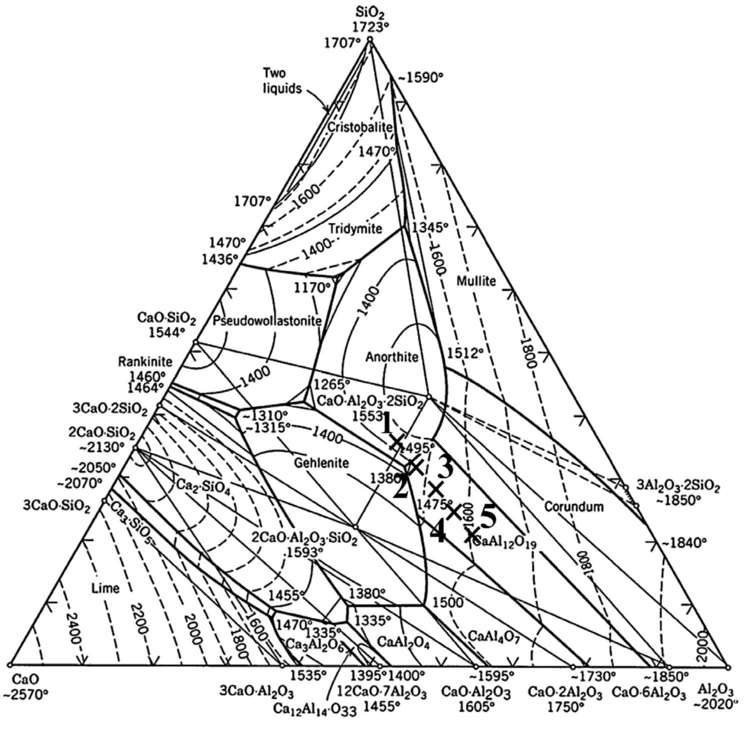

Figure 1. Positions of the compositions FC1-FC5 in the $CaO-Al_2O_3-SiO_2$ phase equilibrium diagram (1 = FC1, 2 = FC2, 3 = FC3, 4 = FC4, and 5 = FC5)

was investigated by a dilatometer (402C, Netzsch-Geratebau GmbH, Selb, Germany). The crystalline phases of the samples were identified by using x-ray diffraction (D8-Advance, Bruker Corp., Billerica, MA, USA) with Cu-K_α radiation, and diffraction data were collected over a 2θ range from 10 to 80^o with a step size of 0.02^o. The semi-quantitative calculationof the phase composition was determined by TOPAS Technical software (Bruker Corp., Coventry, UK).

The thermal expansion coefficient of the fired specimens was calculated from the expansion curves in the temperature range of 30 to 800°C with a heating rate of 10°C/min usinga dilatometer (402C, Netzsch-Geratebau GbmH). The microstructure of the fired specimens wasobserved by a scanning electron microscope(SEM, JSM 6480 LV, JEOL Ltd., Tokyo, Japan).

Results and Discussion

Table 3 showsthe bulk density, water absorption, and appearance porosityof the different compositionsfired at 1000, 1050, 1100, 1150, 1200, and 1250°C. The bulk density of all compositions was slightly decreased with the firing temperature increased. Because of the reaction of the higher density phases, corundum (3.98 g/cm^3), quartz (2.65 g/cm^3), and gehlenite (2.97g/cm^3) changed to the lower density anorthite (2.74 g/cm^3), according to the ternary phase diagram CaO-Al_2O_3-SiO_2 (Traoré *et al.*, 2003). The composition FC3 exhibited the lowest bulk density value at all firing temperatures. Particularly, the lowest value at 1.36 g/cm^3 was achieved after sintering at 1250°C. On the other hand, the apparent porosity and water absorption were decreased with the firing temperature increased with the exception ofcomposition FC3. At 1250°C, the highest water absorption of the samples was around 38.52%. Moreover, the apparent porosity and water absorption increased with a decreasing amount of dolomite clay because of the amount of organic matter and carbonate in dolomite clay.

Total linear shrinkage of the fired samples is shown in Figure 2. The shrinkage increased with increasing the amount of dolomite clay and slightly decreased with increasing the firing temperature. In addition, from the firing behavior of green samples in the temperature range 30-1250°C, as shown in Figure 3, it was seenthat the shrinkage increased with increasing the amount of dolomite clay. The FC3 sample was selected as the optimum composition when the lowest bulk density was considered.

Figure 4 shows the X-ray diffraction patterns of the FC3 green body whenfired at 1000, 1050, 1100, 1150, 1200, and 1250°C. Corundum, quartz, kaolinite, portlandite, calcite, and dolomite phases were identified in the green sample. After firing at 1000°C, anorthite, gehlenite, wollastonite, and calcium silicate phases could be found. After firing at 1200°C, quartz and wollastonite disappeared. At higher temperatures (1200-1250°C), anorthiteand gehlenite were observed as the main phases andcorundum was the minor phase. After calculationof the amount of phasesin the samples which were firedat 1200 and 1250°C usingthe TOPAS Technical software, anorthite was slightly increased with increasing the firing temperature to 1200 and 1250°C (44.89 and 45.96%, respectively).

Table 4 shows the thermal expansion coefficientscalculated in the temperature range of 30-800°C ofsample FC3 fired at 1000, 1050, 1100, 1150, 1200, and 1,250°C. The thermal expansion coefficientswere decreased with increasing the firing temperature because the amount of the anorthite phase was increased (4.9×10^{-6}°C^{-1}), whichagreed with the report of Cheng *et al.* (2012), whereas the higher thermal expansion coefficient phases,gehlenite (28.3×10^{-6}°C^{-1}) (Merlini *et al.*, 2005), wollastonite (6.5×10^{-6}°C^{-1}) (Hans *et al.*, 2009), and corundum (8.2×10^{-6}°C^{-1}) (Aksel, 2003) weredecreased.

Figure 5 shows SEM micrographs of

Table 3. Bulk density, apparent porosity, and water absorption of the compositions FC1-FC5 after firing at different temperatures

Compositions	Temperature (°C)	Properties		
		Bulk density (g/cm^3)	Apparent porosity (%)	Water absorption (%)
FC1	1000	1.46±0.008	51.99± 0.10	35.68± 0.06
	1050	1.45±0.004	51.31± 0.30	35.42± 0.28
	1100	1.45±0.002	51.15± 0.01	35.31± 0.04
	1150	1.45±0.001	50.96± 0.14	35.21± 0.08
	1200	1.45±0.003	50.35± 0.00	34.71± 0.43
	1250	1.43±0.008	48.36± 0.06	33.78± 0.10
FC2	1000	1.46±0.003	52.13± 0.35	35.64± 0.29
	1050	1.45±0.004	51.79± 0.46	35.82± 0.40
	1100	1.44±0.009	51.74± 0.36	35.82± 0.41
	1150	1.44±0.013	51.43± 0.22	35.81± 0.20
	1200	1.42±0.003	50.38± 0.18	35.41± 0.08
	1250	1.41±0.003	49.16± 0.29	34.99± 0.17
FC3	1000	1.45±0.005	53.97± 0.04	37.30± 0.11
	1050	1.44±0.003	53.52± 0.04	37.23± 0.23
	1100	1.44±0.005	53.76± 0.09	37.44± 0.04
	1150	1.43±0.001	52.86± 0.06	37.05± 0.03
	1200	1.40±0.007	51.59± 0.02	36.78± 0.12
	1250	1.36±0.065	52.30± 1.88	38.52± 2.67
FC4	1000	1.47±0.002	54.07± 0.05	36.70± 0.01
	1050	1.47±0.001	53.66± 0.01	36.54± 0.01
	1100	1.45±0.004	53.73± 0.68	37.03± 0.13
	1150	1.45±0.003	53.30± 0.09	36.76± 0.12
	1200	1.43±0.005	51.58± 0.25	36.18± 0.27
	1250	1.44±0.005	50.95± 0.44	35.49± 0.39
FC5	1000	1.49±0.009	54.96± 0.13	36.92± 0.13
	1050	1.44±0.001	55.22± 0.11	37.57± 0.88
	1100	1.45±0.005	55.14± 0.17	37.77± 0.20
	1150	1.46±0.001	54.40± 0.11	37.44± 0.09
	1200	1.47±0.008	52.48± 0.18	36.36± 0.27
	1250	1.49±0.008	52.73± 0.10	36.36± 0.22

the fracture surface of sample FC3 fired at 1000°C (Figure 5(a)), 1050°C (Figure 5(b)), 1100°C (Figure 5(c)), 1150°C (Figure 5(d)), 1200°C (Figure 5(e)), and 1250°C (Figure 5(f)), respectively. The connectionbetween the grains and the average grain sizes are increased with increasing the firing temperature. The un-reacted quartz crystalsstill remain in the samplesafter being fired at 1000-1150°C as indicated in Figure 5(a) to 5(d) with arrows. Crystals of quartz disappeared at firing temperaturesof 1200°C (Figure 5(e)) and 1250°C (Figure 5(f)).

Conclusions

In this work, the mixture of dolomite clay, calcined alumina, and white portland cement (40%, 35%, and 25%, respectively) fired at 1250°C exhibited anorthite and gehlenite phases. Moreover, the samples had low density, high porosity, and high water absorption (1.36 g/cm^3, 52.30%, and 38.52%, respectively). On the other hand, the thermal

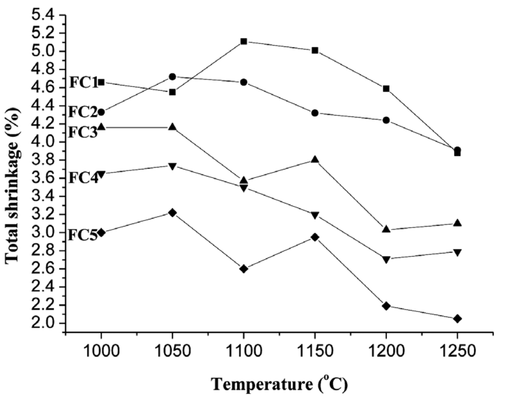

Figure 2. Total shrinkage of different composition samples after firing at different temperatures

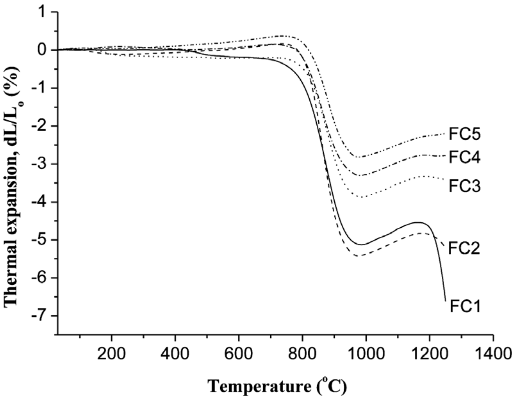

Figure 3. Firing behavior of the samples in the temperature range 30-1250°C

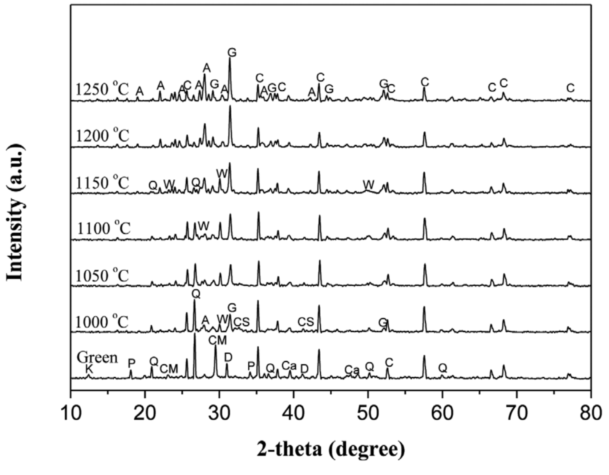

Figure 4. XRD spectrum of FC3 samples fired at different temperatures: (A = anorthite, C = corundum, G = gehlenite, Q = quartz, W = wollastonite, CS = calcium silicate, K = kaolinite, P = portlandite, Ca = calcite, D = dolomite)

expansion coefficient of the samplesstill had a high value at 7.09×10^{-6}°C^{-1}. The composition will be changed to produce only anorthiteas a single phase for decreasing the thermal expansion coefficient, which will be reported in a further study.

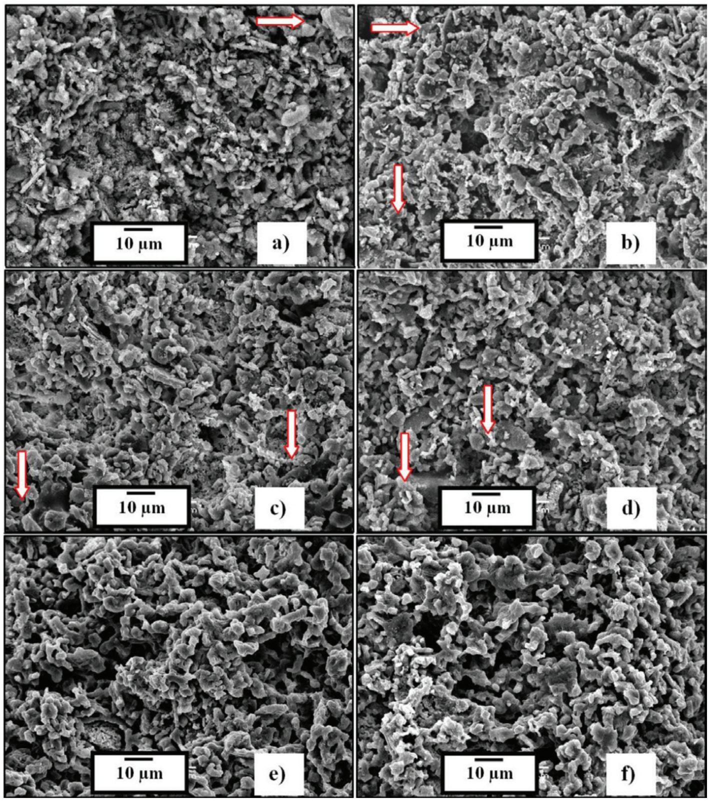

Figure 5. SEM micrographs of the fracture surface of sample FC3 fired at different temperatures: a) 1000°C, b) 1050°C, c) 1100°C, d) 1150°C, e) 1200°C, and f) 1250°C; the arrows indicate crystals of the un-reacted quartz

Table 4. Thermal expansion coefficients calculated in the temperature range 30-800°C of FC3 samples after firing at different temperatures

Temperature (°C)	Thermal expansion coefficients (×10^{-6}°C^{-1})
1000	8.78
1050	8.52
1100	8.33
1150	7.73
1200	7.13
1250	7.09

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