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Trans. Nonferrous Met. Soc. China 22(2012) 3108-3112

Transactions of Nonferrous Metals Society of China

www.tnmsc.cn

# Effect of Na<sub>2</sub>O on formation of calcium aluminates in CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system

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Received 12 December 2011; accepted 21 May 2012

Abstract: The formation characteristics of calcium aluminates in the CaO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> system with sodium oxide was investigated by XRD, SEM–EDS and DSC–TG technologies. The main phases in the clinker after sintering at 1350 °C are 12CaO·7Al<sub>2</sub>O<sub>3</sub>, 2CaO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> and 2CaO·SiO<sub>2</sub> when the mass ratio of Al<sub>2</sub>O<sub>3</sub> to SiO<sub>2</sub> is 3.0 and the molar ratio of CaO to Al<sub>2</sub>O<sub>3</sub> is 1.0. The proportion of 12CaO·7Al<sub>2</sub>O<sub>3</sub> increases with the increase of Na<sub>2</sub>O addition when the molar ratio of Na<sub>2</sub>O to Al<sub>2</sub>O<sub>3</sub> is from 0 to 0.4, while the proportion of 2CaO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> decreases with the increase of Na<sub>2</sub>O addition. Na<sub>2</sub>O forms solid solution in 12CaO·7Al<sub>2</sub>O<sub>3</sub>, which increases the volume of elementary cell of 12CaO·7Al<sub>2</sub>O<sub>3</sub>. The formation temperature of 12CaO·7Al<sub>2</sub>O<sub>3</sub> is decreased by 30 °C when the molar ratio of Na<sub>2</sub>O to Al<sub>2</sub>O<sub>3</sub> increases from 0 to 0.4 determined by DSC. The alumina leaching property of clinker increases obviously with the increase of Na<sub>2</sub>O addition.

Key words: CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system; Na<sub>2</sub>O; Al<sub>2</sub>O<sub>3</sub>; 12CaO·7Al<sub>2</sub>O<sub>3</sub>; sintering; leaching

# **1** Introduction

CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> (C-A-S) ternary system is of considerable importance in oxide ceramics, cement chemistry, metallurgical slags and geochemistry [1–3]. Meanwhile, the C-A-S system plays an important role in the production of alumina industry by the sintering process in China [4]. The phase diagram of the C-A-S system can be seen elsewhere, and the thermodynamic analyses of the ternary system have been performed by PELTON et al [5] using a quasichemical model, by WANG et al [6] using an ionic sublattice model and by FABRICHNAYA and NERAD [7] using a molecular model.

Meanwhile, the formation characteristics of calcium aluminates has been widely investigated [8,9], such as  $3CaO \cdot Al_2O_3$  (C<sub>3</sub>A) [10],  $12CaO \cdot 7Al_2O_3$  (C<sub>12</sub>A<sub>7</sub>) [11], CaO \cdot Al\_2O\_3 (CA) [12], CaO · 2Al<sub>2</sub>O<sub>3</sub> (CA<sub>2</sub>) [13] and CaO · 6Al<sub>2</sub>O<sub>3</sub> (CA<sub>6</sub>) [14]. GRZESZCZYK [15] and OSTROWSKI and FLAZNY [16] studied the solid solutions of calcium aluminates with sodium ion formed at high temperature in CaO-Al<sub>2</sub>O<sub>3</sub> binary system, and sodium ions can be built into the calcium aluminate system. Furthermore, SUN et al [17] found that addition of Na<sub>2</sub>O during the sintering process can promote the alumina leaching property of calcium aluminate slag, but the mechanism was not proposed.

In the C-A-S system, the phases in the sintered clinker referred to alumina production consist of calcium aluminates,  $2CaO \cdot SiO_2$  (C<sub>2</sub>S) and  $2CaO \cdot Al_2O_3 \cdot SiO_2$ (C<sub>2</sub>AS). C<sub>2</sub>S comprises several different types, such as  $\alpha'$ -C<sub>2</sub>S,  $\beta$ -C<sub>2</sub>S,  $\gamma$ -C<sub>2</sub>S. Previous studies [18] indicated that  $C_{12}A_7$  has better alumina leaching properties than other calcium aluminates in sodium carbonate solution, while  $C_2AS$  is difficult to be extracted to solution.  $C_2S$  is very stable in sodium carbonate solution. For the great role of sodium oxide during the sintering process, it is necessary to reveal the reaction mechanisms in the C-A-S system in the presence of sodium ions. Therefore, the aim of this work is to study the effect of sodium oxide on the sintering characteristics and leaching property of calcium aluminates in the C-A-S system with low mass ratios of  $Al_2O_3$  to  $SiO_2$  and CaO to  $Al_2O_3$ .

# 2 Experimental

Analytically pure reactants were used in the present

Foundation item: Projects (51174054, 51104041) supported by the National Natural Science Foundation of China Corresponding author: YU Hai-yan; Tel: +86-24-83686460; E-mail: yuhy@smm.neu.edu.cn DOI: 10.1016/S1003-6326(11)61578-1

work and the calculated oxide ratios for sintering process are listed in Table 1. CaO and Na<sub>2</sub>O were added in the forms of CaCO<sub>3</sub> and Na<sub>2</sub>CO<sub>3</sub>, respectively. The mass ratio of Al<sub>2</sub>O<sub>3</sub> to SiO<sub>2</sub> (A/S) is 3.0, and the molar ratio of CaO to Al<sub>2</sub>O<sub>3</sub> (C/A) is 1.0 (the residual CaO subtracted the composition to form 2CaO·SiO<sub>2</sub>). The molar ratio of Na<sub>2</sub>O to Al<sub>2</sub>O<sub>3</sub> (N/A) ranges from 0 to 0.4 as presented in Table 1. The mixtures were milled in a ball mill for 3 h, and then sintered at 1350 °C for 1 h in a MoSi<sub>2</sub> resistance furnace followed by cooling in the furnace.

Table 1 Oxide ratios of samples for sintering

			-	-	
N/	A w(A	Al <sub>2</sub> O <sub>3</sub> )/%	w(CaO)/%	w(SiO <sub>2</sub> )/%	w(Na <sub>2</sub> O)/%
(	)	39.93	46.36	13.31	0
0.	1	38.98	45.66	12.99	2.37
0.	2	38.08	44.60	12.69	4.63
0.	3	37.22	43.59	12.41	6.79
0.	4	36.39	42.63	12.13	8.85

The sintered clinkers were leached at 75 °C for 30 min in sodium carbonate solution. The concentration of sodium carbonate solution (in the form of Na<sub>2</sub>O) is 80 g/L. The liquid-to-solid ratio of sodium carbonate solution to clinker for leaching is 10. The leached slurry was filtrated using a Buchner funnel. The concentration of caustic alkali (N<sub>K</sub>), total alkali (N<sub>T</sub>) and Al<sub>2</sub>O<sub>3</sub> (AO) in the filter liquor were determined by the volumetric method, while the filter residue was washed carefully and dried for chemical analysis. The alumina leaching rate is calculated according to the following formula:

$$\eta_{\rm Al_2O_3} = \left(1 - \frac{(\rm A/S)_{\rm residue}}{(\rm A/S)_{\rm clinker}}\right) \times 100\%$$
(1)

where  $(A/S)_{residue}$  and  $(A/S)_{clinker}$  are the mass ratios of  $Al_2O_3$  to  $SiO_2$  in the leached residue and sintered clinker, respectively.

The contents of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and Na<sub>2</sub>O in samples and filtrate were analyzed by X-ray fluorescence (XRF, ZSX100e). Phase components of the clinker were identified by X-ray diffraction (PANalytical PW3040/60). SEM (SHIMADZU SSX–550) and EDS (DX-4) were used for microstructure and component analysis. Simultaneously recorded studies of differential scanning calorimetry (DSC) and thermogravimetric analysis were carried out using a NETZSCH STA409C/CD simultaneous thermal analyzer in a dynamic Ar atmosphere. The samples with the N/A ratios of 0 and 0.4 in Table 1 were selected. The samples were heated up to 1500 °C at a rate of 10 °C/min.

# **3** Results and discussion

#### 3.1 Phase composition characteristics

The XRD patterns of sintered clinkers at different N/A ratios are shown in Fig. 1. When N/A=0.1, the clinker contains C<sub>2</sub>AS, C<sub>12</sub>A<sub>7</sub>,  $\beta$ -C<sub>2</sub>S,  $\gamma$ -C<sub>2</sub>S as well as some CA and CA<sub>2</sub>. As the N/A ratio increases to 0.2, the phase composition and content of clinker are different. The content of C<sub>12</sub>A<sub>7</sub> increases, while the content of C<sub>2</sub>AS decreases. Meanwhile, CA<sub>2</sub> disappears, and  $\beta$ -C<sub>2</sub>S is formed during the sintering process. When N/A=0.4, most of the clinker is C<sub>12</sub>A<sub>7</sub>, and the content of C<sub>2</sub>AS is very low. Furthermore, both CA and CA<sub>2</sub> do not exist, while  $\beta$ -C<sub>2</sub>S and  $\gamma$ -C<sub>2</sub>S coexist in the clinker. It can be concluded that as the molar ratio of Na<sub>2</sub>O to Al<sub>2</sub>O<sub>3</sub> increases from 0 to 0.4, the proportion of C<sub>12</sub>A<sub>7</sub> in the clinker increases with the increase of N/A ratio when A/S=3.0 and C/A=1.0.



Fig. 1 XRD patterns of sintered clinkers at different N/A ratios

The crystal structure of  $C_{12}A_7$  also changes as the N/A ratio increases. The interplanar spacing corresponding to the strongest characteristic peak  $(2\theta=18.109^\circ)$  of  $C_{12}A_7$  and the parameter of cubic cell as well as its volume at different N/A were calculated, as listed in Table 2. Both the interplanar spacing and the volume of elementary cell of  $C_{12}A_7$  increase with the

	Table 2	Effect of N/A	ratio on lattice	parameters of $C_{12}A_7$
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N/A	Interplanar	Parameter of	Volume of cubic
N/A	spacing, d/Å	cubic cell, a/Å	cell/Å <sup>3</sup>
0	4.8945	11.9890	1723.25
0.1	4.8950	11.9968	1726.62
0.2	4.8967	11.9979	1727.09
0.3	4.8975	12.0035	1729.51
0.4	4.8980	12.0090	1731.89

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increase of N/A, indicating that Na<sub>2</sub>O forms solid solution in  $C_{12}A_7$ , which is consistent with Ostrowski's results [16]. Therefore, the solid solution of Na<sub>2</sub>O in  $C_{12}A_7$  is beneficial to the formation of  $C_{12}A_7$ .

When N/A=0.4, the representative microstructure of sintered clinker is shown in Fig. 2. The morphology of particles is massive, and the particles can be divided to two kinds by the size. Most of the particles have a larger size, and are usually several micrometers, even larger than 10  $\mu$ m. The other particles are relatively small, and are usually smaller than 1  $\mu$ m. These particles are adsorbed on the surface of the larger particles.



**Fig. 2** SEM images of sintered clinker when N/A=0.4: (a) Backscattered electron image; (b) Secondary electron image

The compositions of larger particles (points 1 and 2 in Fig. 2(b)) and smaller particles (point 3) determined by EDS analysis are listed in the left of Table 3, and the corresponding compositions of oxides are calculated in the right of Table 3. No SiO<sub>2</sub> is discerned in the large particles, and the compositions are similar to of C<sub>12</sub>A<sub>7</sub>. Therefore, the larger particles are  $C_{12}A_7$ . The composition ratio of CaO to SiO<sub>2</sub> in the smaller particles is close to 2, indicating that the smaller particles are  $C_2S$ . Furthermore, as presented in Table 3, the composition of Na<sub>2</sub>O in the larger particles is about 4%, while the composition of Na<sub>2</sub>O in the smaller particles is below 1%. Because no compound containing Na<sub>2</sub>O was found by XRD analysis, Na<sub>2</sub>O must be solid dissolved in the crystal lattice of  $C_{12}A_7$ , which is consistent with the calculated results of lattice parameters of C12A7, as presented in Table 2.

 Table 3 Phase compositions of clinker when N/A=0.4 corresponding to Fig. 2(b)

		<u> </u>	)	-1			Cala	المغمط	
Doint	Original composition/%				Calculated				
Font	0	Na	A 1	C	<b>C</b> :	No O	A1.0	C-O	<u>s:0</u>
	0	INa	AI	Ca	51	Na <sub>2</sub> O	$AI_2O_3$	CaO	5102
1	38.46	2.72	27.45	31.37	0.00	3.67	51.85	34.73	0.00
2	39.03	3.06	27.87	30.04	0.00	4.12	52.65	33.26	0.00
3	38.23	0.60	20.85	30.86	9.47	0.81	39.38	34.16	20.30

#### 3.2 Thermal analysis

The DSC heating and TG curves of both samples with the N/A ratios of 0 and 0.4 are shown in Fig. 3. Two strong endothermic peaks with a large gravity decrease at 798 °C exist in both curves, indicating that they relate to the decomposition of CaCO<sub>3</sub>. A small endothermic peak with a small gravity decrease at 864 °C in Fig. 3(b) indicates that it relates to the melting of Na<sub>2</sub>CO<sub>3</sub>. C<sub>2</sub>S is formed below 1300 °C [4], occurring at the endothermic peaks between 1000 °C and 1300 °C for both samples. As shown in Fig. 1, C<sub>12</sub>A<sub>7</sub> is the main phase in the clinker when the N/A ratio is 0.4, and therefore, the large endothermic peak at 1360 °C in Fig. 3(b) relates to the



**Fig. 3** DSC-TG curves of different mixtures during heating process: (a) N/A=0; (b) N/A=0.4

formation of  $C_{12}A_7$ . As an intermediate phase,  $C_2AS$  is formed before  $C_{12}A_7$  in the CaO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> system [4]. Therefore, the endothermic peak at 1342 °C should be associated with the formation of  $C_2AS$ , and the endothermic peak at 1390 °C represents the formation of  $C_{12}A_7$ . Addition of Na<sub>2</sub>O can not only promote the formation of  $C_{12}A_7$  and inhibit the formation of  $C_2AS$ , but also decrease the formation temperature of  $C_{12}A_7$  by 30 °C.

#### 3.3 Alumina leaching property

The leaching results of clinkers at different N/A ratios in sodium carbonate solution are listed in Table 4, and alumina leaching rates are calculated by formula (1) as shown in Fig. 4. The reaction of calcium aluminates with sodium carbonate solution is shown in formula (2).

$$x \text{CaO·yAl}_2\text{O}_3 + x \text{Na}_2\text{CO}_3 + (x+3y)\text{H}_2\text{O} \longrightarrow$$
$$x \text{CaCO}_3 \downarrow + 2y \text{NaAl}(\text{OH})_4 + (2x-2y) \text{NaOH}$$
(2)

The alumina leaching rate increases obviously with the increase of N/A ratio. It is 86.91% when N/A=0.4, which is much higher than that when N/A=0 by 43.52%. Therefore, the addition of Na<sub>2</sub>O can greatly improve the leaching properties of sintered clinker.

The XRD patterns of leached residues are shown in Fig. 5. The main phases are CaCO<sub>3</sub>,  $C_2S$  and  $C_2AS$ . No

 Table 4 Leaching results of clinkers at different N/A ratios in sodium carbonate solution

N/A	Liquor co	ncentration	Solid composition/%		
	$N_K$	$N_{\mathrm{T}}$	AO	$Al_2O_3$	SiO <sub>2</sub>
0	15.32	89.69	18.16	23.06	13.58
0.1	20.15	89.35	22.90	17.49	14.03
0.2	24.18	90.08	26.71	13.33	14.01
0.3	28.81	90.12	31.51	8.24	14.86
0.4	30.43	89.80	33.68	5.71	14.54



Fig. 4 Effect of N/A ratio on alumina leaching rate of clinkers



Fig. 5 XRD patterns of leached residues at different N/A ratios

 $C_{12}A_7$  is found in the leached residues, indicating that all  $Al_2O_3$  in  $C_{12}A_7$  is extracted into the solution during the leaching process.

# 4 Conclusions

1) Na<sub>2</sub>O promotes the formation of  $C_{12}A_7$  and inhibits the formation of  $C_2AS$  sintered at 1350 °C when A/S=3.0 and C/A=1.0 in the CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system.

2) Na<sub>2</sub>O forms solid solution in  $C_{12}A_7$  which increases the volume of elementary cell of  $C_{12}A_7$ .

3) The formation temperature of  $C_{12}A_7$  is decreased by 30 °C when the molar ratio of Na<sub>2</sub>O to Al<sub>2</sub>O<sub>3</sub> increases from 0 to 0.4, and the leaching properties of the sintered clinker are greatly increased.

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# 氧化钠对 CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> 三元系铝酸钙形成规律的影响

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摘 要:利用 XRD、SEM-EDS 和 DSC-TG 技术研究了添加 Na<sub>2</sub>O 的 CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> 体系中铝酸钙的形成规律。 结果表明,当 Al<sub>2</sub>O<sub>3</sub>与 SiO<sub>2</sub> 的质量比为 3.0、CaO 与 Al<sub>2</sub>O<sub>3</sub> 的摩尔比为 1.0 时,在 1350 ℃ 烧结后的熟料主要由 12CaO·7Al<sub>2</sub>O<sub>3</sub>、2CaO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> 和 2CaO·SiO<sub>2</sub> 组成。熟料中 12CaO·7Al<sub>2</sub>O<sub>3</sub> 的含量随着 Na<sub>2</sub>O 的增加而增加, 2CaO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> 的含量随着 Na<sub>2</sub>O 的增加而降低。Na<sub>2</sub>O 在 12CaO·7Al<sub>2</sub>O<sub>3</sub> 中形成固溶体,增加了其单位晶胞体积。 DSC 分析表明,Na<sub>2</sub>O 不仅促进了 12CaO·7Al<sub>2</sub>O<sub>3</sub>的形成,而且使 C<sub>12</sub>A<sub>7</sub>的形成温度降低了 30 ℃。烧结熟料中的 氧化铝溶出性能随着 Na<sub>2</sub>O 的增加而大幅度提高。

关键词: CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>系; Na<sub>2</sub>O; Al<sub>2</sub>O<sub>3</sub>; 12CaO·7Al<sub>2</sub>O<sub>3</sub>; 烧结; 溶出

(Edited by YANG Hua)