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INFLUENCE OF THE COMPOSITION AND COOLING RATE OF ALUMOCALCIUM SLAG ON ITS CRUMBLABILITY

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Abstract. The main components of metallurgical slags are iron compounds, which are extracted by reduction smelting. The process of obtaining various products based on iron and slags of different compositions (alumocalcium self-crumbling, etc.) can be implemented in several ways. It is important to use a mode of smelting and cooling of the alumocalcium slag formed during melting in the furnace that ensures its most complete spontaneous crumbling and high rates of extraction of REM from it. Synthetic slags having a phase composition similar to industrial samples after the smelting of iron ores were selected for the experiments. The simulated samples correspond to the dicalcium silicate primary crystallisation region on the ternary phase diagram of the $CaO–SiO_2–Al_2O_3$ system. After crumbling, the slag was subjected to sieve analysis using a mechanical sieve. Slags with a silicon modulus $k = 2.0$ that actively crumbled during cooling were used in the experiments. A higher silicon modulus results in a lower crumblability. It was established that it is impossible to precisely limit the composition areas of the crumbling slags at specific cooling rates. The studies showed that the crumblability of slags improves when moving towards the centre of the dicalcium silicate region. The composition of the slags is close to the composition of the points located in the area bounded by the lines $2CaO·SiO_2 – 2CaO·Al_2O_3$ and $2CaO·SiO_2 – 12CaO·7Al_2O_3$ on one side and by the lines of the silicon modulus no higher than 2.85 – 3.00 on the other side. The granulometric composition is almost independent of the cooling rate. The temperature mode from smelting to cooling affects the crumblability of the slags. The most promising are slags with a silicon modulus in the range of 2.85 – 3.00 close to the phase triangle $12CaO·7Al_2O_3 – 2CaO·SiO_2 – 2CaO·Al_2O_3$.

Keywords: metallurgical slag, alumocalcium silicate, cooling rate, slag crumblability, silicon module, sieve analysis, granulometric composition

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Оригинальная статья

ВЛИЯНИЕ СОСТАВА И СКОРОСТИ ОХЛАЖДЕНИЯ АЛЮМОКАЛЬЦИЕВОГО ШЛАКА НА ЕГО РАССЫПАЕМОСТЬ

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Аннотация. Основным компонентом металлургических шлаков являются соединения железа, которые извлекаются проведением восстановительной плавки. Известно несколько типов данного процесса с получением различных продуктов на основе железа и шлаков разного состава (алюмокальциевого самомрассыпающегося и др.). Режим плавки и охлаждения образованного в процессе плавки в печи алюмокальциевого шлака должен обеспечивать наиболее полное самопроизвольное его рассыпание, а также высокие показатели извлечения из него редкоземельных металлов. Для опытов в работе выбраны синтетические шлаки, схожие по фазовому составу с промышленными образцами после выплавки железосодержащих руд. Смоделированные образцы соответствуют области первичной кристаллизации двухкальциевого силиката на тройной диаграмме состояния системы $CaO–SiO_2–Al_2O_3$. Шлак после рассыпания подвергали ситовому анализу с помощью механического сита. В опытах использовались шлаки с кремниевым модулем $k = 2,0$, которые активно рассыпалась в момент их охлаждения. При увеличении кремниевого модуля рассыпаемость ухудшается. Установлено, что точно ограничить области составов рассыпающихся шлаков при определенных скоростях охлаждения невозможно. Проведенные исследования показали, что рассыпаемость шлаков улучшается по мере приближения к центру области двухкальциевого силиката. Состав шлаков близок к составу точек, расположенных в области, ограниченной с одной стороны линиями $2CaO·SiO_2 – 2CaO·Al_2O_3$ и $2CaO·SiO_2 – 12CaO·7Al_2O_3$, и с другой стороны линиями кремниевого модуля не выше 2,85 – 3,00. При этом гранулометрический состав почти не зависит от скорости охлаждения. На рассыпаемость шлаков влияет температурный режим от выплавки до охлаждения. Наиболее перспективными являются шлаки с кремниевым модулем в пределах 2,85 – 3,00, близкие к фазовому треугольнику $12CaO·7Al_2O_3 – 2CaO·SiO_2 – 2CaO·Al_2O_3$.

Ключевые слова: металлургический шлак, алюмокальциевый силикат, скорость охлаждения, рассыпаемость шлака, кремниевый модуль, ситовой анализ, гранулометрический состав

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Introduction

The proposed workflow for the complex processing of bauxite raw materials [1 – 3] in rotary kilns involves the production of self-crumbling alumocalcium slags suitable for extracting rare-earth metals (REMs) from them [4, 5] and the transfer of iron into separate slag phase [6, 7].

The mode of heat treatment and subsequent cooling of the slag discharged from the furnace must ensure its complete spontaneous crumbling [8 – 10], which could contribute to the extraction of valuable components such as titanium, scandium, yttrium, etc., from it [11, 12]. The most important technological factors determining phase transformations in slag are its chemical composition and cooling rate [13 – 15].

Alternative methods for extracting REMs are being developed for raw materials with a high SiO_2 content [16 – 18]. Slags with a silicon modulus of 2.00 are typical for complex processing and most clearly demonstrate changes in the granulometric composition after self-crumbling [19]. Complete self-crumbling of the sample can be achieved by selecting the heat treatment and cooling modes individually for each composition [20, 21]. In this case, the stability of the system, which can be estimated from the ternary phase diagram, plays a crucial role [22, 23].

Under these restrictions, slags with a silicon modulus of 2.85 are expected to not crumble when cooled at a rate of 30 °C/min. However, in the experiments carried out in [24, 25], part of the slags with the specified silicon modulus crumbled easily at the suggested rate. This demonstrates that the region in which the crumbling occurs is limited by individual conditions for each sample (the ratio of phases and the modulus) [26, 27]. The result of the crumbling depends on the thermal mode (sintering temperature, holding time, and cooling rate of the composition) [28].

Research methods

In this work, synthetic slags with a phase composition corresponding to the composition of real slags obtained from smelting ferruginous high-silica bauxites and iron ores were used [29]. The selected slag compositions on the ternary phase diagram of the $CaO-SiO_2-Al_2O_3$ system correspond to the dicalcium silicate primary crystallisation region [30 – 32]. Fig. 1 shows the compositions of slags with different silicon moduli (Al_2O_3/SiO_2) [33 – 35]. After cooling, slags smelted in a graphite crucible in the Tamman furnace were crumbled or crushed, then mixed thoroughly. A 70 g subsample was taken from the averaged sample, after which the samples were placed in a graphite crucible of a special design and heated to 1500 °C.

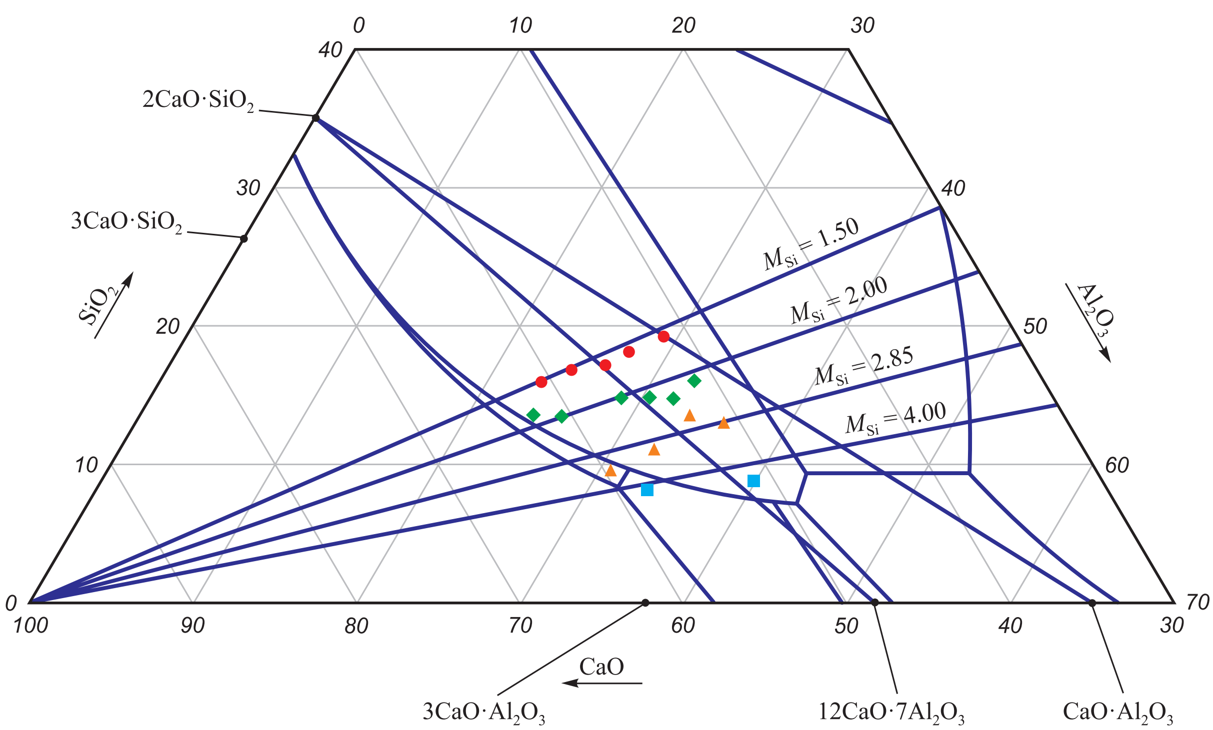

Fig. 1. Compositions of the studied synthetic slags:
● – slags with a silicon module of 1.50; ◆ – slags with a silicon module of 2.00;
▲ – slags with a silicon module of 2.85; ■ – slags with a silicon module of 4.00 [38 – 40]

Рис. 1. Составы исследованных синтетических шлаков:
● – шлаки с кремниевым модулем 1,50; ◆ – шлаки с кремниевым модулем 2,00;
▲ – шлаки с кремниевым модулем 2,85; ■ – шлаки с кремниевым модулем 4,00 [38 – 40]

Table 1

Granulometric composition of slags with a silicon modulus of 2.0 after cooling, %

Таблица 1. **Гранулометрический состав шлаков с кремниевым модулем 2,0 после охлаждения, %**

Slag composition, %	Grade, mm	Cooling rate, °C/min			
		3	7	15	30
CaO = 51.5; SiO_2 = 15.5; Al_2O_3 = 33.0	+0.400	–	23.6	23.4	–
	0.400 – 0.315	–	13.1	8.6	–
	0.315 – 0.200	0.4	22.9	23.0	12.6
	0.200 – 0.160	1.4	8.8	7.7	13.3
	0.160 – 0.100	13.4	9.4	10.2	21.7
	0.100 – 0.063	30.8	9.0	10.4	21.1
	0.063 – 0.050	14.9	3.8	5.0	9.5
	–0.050	39.1	9.6	11.8	21.4
Total		100.0	100.0	100.0	100.0

The melting point of ferroalloys can be lowered by introducing such elements as aluminium into them [36, 37].

After holding at a set temperature for 15 min, the slags were cooled at the rates given below to a temperature of 900 °C, held for 15 min, then removed from the furnace. The slags were further cooled in open air to room temperature. After crumbling, the slags underwent sieve analysis on a set of standard Retsch AS 200 sieves. The ternary phase diagram shows the regions of slag compositions bounded by the lines of different silicon moduli (Al_2O_3/SiO_2). The furnace temperature was controlled automatically with an accuracy of ±5 °C.

Results

Table 1 presents the results of sieve analyses of cooled slags with a silicon modulus of 2.0. Specific slags with a silicon modulus of 2.0 were used to demonstrate the patterns obtained.

In this work, the effect of different factors on the crumblability of slags was studied. The cooling rate and the chemical (hence, phase) composition of slags significantly affect their crumblability. The phase diagram of the $CaO–SiO_2–Al_2O_3$ system had regions of compositions of slags, the spontaneous decomposition of which required specific cooling rates [41 – 43].

The cooling rate of 2 °C/min from the liquidus temperature to 1000 °C ensures the crumbling of all slags in the bicalcium silicate crystallisation region. Slags containing 6 – 9 % SiO_2, 52 – 58 % CaO, 30 – 37 % Al_2O_3 cooled at a rate of 7 °C/min crumble in the temperature range from the liquidus to 1000 °C. Slags containing 9 – 14 % SiO_2, 47 – 60 % CaO, 30 – 40 % Al_2O_3 crumble upon cooling at a rate of 15 °C/min in the same temperature range, and slags containing 14 % SiO_2, 51 – 60 % CaO – in the temperature range from the liquidus to room temperature. Fully crumbled slags completely pass through a sieve with a mesh size of 100 µm.

The optimal phase can be obtained with excess amounts of silicon in the initial mixture [44].

It has been established that the melting temperature and the lower cooling limit affect slag crumbling. Table 2 shows the granulometric composition of slag of a similar chemical composition heated to different temperatures (1300, 1350, 1400, and 1600 °C) and cooled at a rate of 30 °C/min to room temperature.

Table 3 shows a sieve analysis of slag having a similar composition cooled from a temperature of 1500 °C to temperatures of 700, 800, 900, 1000, and 1100 °C at a constant controlled rate of 10 °C/min. Next, the samples were

Table 2

Granulometric composition of the slag cooled at the same rate, %

Таблица 2. **Гранулометрический состав шлака, охлажденного с одинаковой скоростью, %**

Grade, mm	Heating temperature, °C			
	1300	1350	1400	1600
+0.400	–	3.1	3.2	5.4
0.400 – 0.315	–	1.1	1.1	0.4
0.315 – 0.200	–	7.2	7.9	5.8
0.200 – 0.160	16.5	16.9	9.4	5.4
0.160 – 0.100	12.3	8.8	3.6	10.5
0.100 – 0.063	19.1	11.7	13.7	18.5
0.063 – 0.050	13.8	12.1	8.3	10.1
–0.050	38.3	39.0	52.5	43.8
Total	100.0	100.0	100.0	100.0

removed from the furnace and further cooled in open air at an arbitrary rate. The temperature gradient (upper and lower values) affects the granulometric composition and the subsequent crumbling [45].

Discussion

Fig. 2 shows a granulometric and chemical composition of the slags.

The studies showed that the crumblability of slags increases when moving from the boundaries of the dicalcium silicate region towards the centre. The composition of the slags is close to the composition of points located in the area bounded on one side by $2CaO{\cdot}SiO_2$ – – $2CaO{\cdot}Al_2O_3$ and $2CaO{\cdot}SiO_2$ – $12CaO{\cdot}7Al_2O_3$ lines, and on the other side, with the silicon modulus lines no higher than 2.85 – 3.00. The granulometric composition of the slag was found to be almost independent of its cooling rate [46].

Approaching the boundaries of the dicalcium silicate region, the cooling rate begins to significantly af-

Table 3

Granulometric composition of the slag cooled at the same rate from 1500 °C to the specified temperatures, %

Таблица 3. **Гранулометрический состав шлака, охлажденного с одинаковой скоростью от 1500 °C до указанных температур, %**

Grade, mm	Cooling temperature, °C				
	700	800	900	1000	1100
+0.400	–	–	–	–	36.4
0.400 – 0.315	–	–	–	–	1.1
0.315 – 0.200	–	–	0.9	0.6	3.6
0.200 – 0.160	–	–	1.3	2.4	2.7
0.160 – 0.100	0.9	0.8	8.9	11.4	9.5
0.100 – 0.063	15.1	15.4	23.6	23.5	11.0
0.063 – 0.050	19.0	18.5	16.3	15.4	8.2
–0.050	65.0	65.8	48.9	46.8	27.9
Total	100.0	100.0	100.0	100.0	100.0

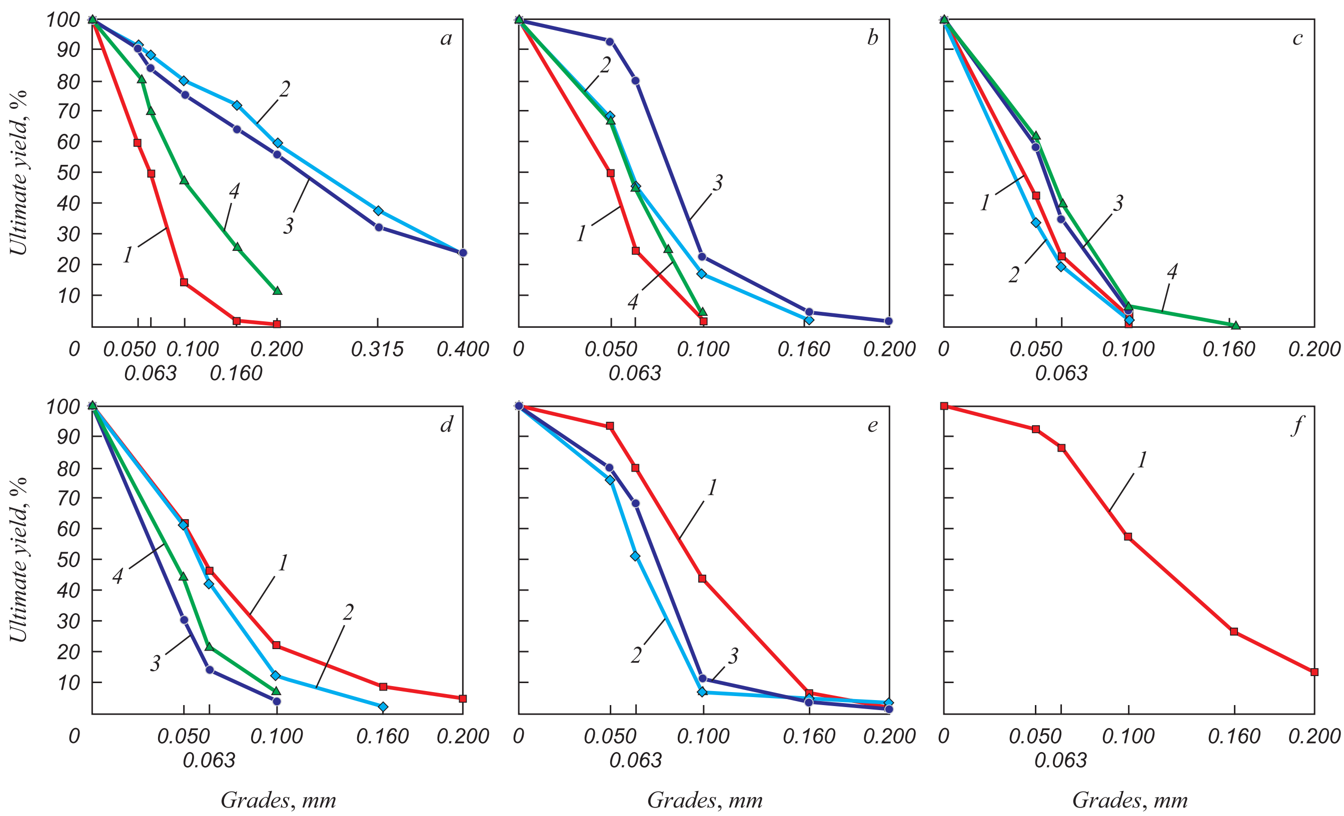

Fig. 2. Granulometric composition of slag containing:
a – 51.5 CaO, 15.5 SiO_2, 33.0 Al_2O_3; *b* – 53.4 CaO, 14.1 SiO_2, 32.5 Al_2O_3; *c* – 56.5 CaO, 14.2 SiO_2, 29.3 Al_2O_3;
d – 54.4 CaO, 14.6 SiO_2, 30.9 Al_2O_3; *e* – 60.5 CaO, 13.2 SiO_2, 26.3 Al_2O_3; *f* – 62.6 CaO, 12.0 SiO_2, 25.4 Al_2O_3;
1 – slags with silicon module 1.50; *2* – slags with silicon module 2.00; *3* – slags with silicon module 2.85;
4 – slags with silicon module 4.00 [36 – 38]

Рис. 2. Гранулометрический состав шлака, содержащего, %:
a – 51,5 CaO, 15,5 SiO_2, 33,0 Al_2O_3; *b* – 53,4 CaO, 14,1 SiO_2, 32,5 Al_2O_3; *c* – 56,5 CaO, 14,2 SiO_2, 29,3 Al_2O_3;
d – 54,4 CaO, 14,6 SiO_2, 30,9 Al_2O_3; *e* – 60,5 CaO, 13,2 SiO_2, 26,3 Al_2O_3; *f* – 62,6 CaO, 12,0 SiO_2, 25,4 Al_2O_3;
1 – шлаки с кремниевым модулем 1,50; *2* – шлаки с кремниевым модулем 2,00; *3* – шлаки с кремниевым модулем 2,85;
4 – шлаки с кремниевым модулем 4,00 [36 – 38]

fect the granulometric composition of the slags, as Fig. 2 clearly illustrates.

The lower the slag cooling rate, the more accurately the crystallisation equilibrium conditions are satisfied. Slag crumblability was expected to improve with a decreasing cooling rate.

After crumbling, slags cooled at a rate of 30 °C/min have a finer granulometric composition than slags of a similar chemical composition cooled at a rate of 7 and 15 °C/min. This phenomenon is probably due to the formation of a larger number of crystal nuclei during rapid cooling [47].

It is also important to note that slag crumblability decreases with an increasing silicon modulus. In the authors' experiments, only one slag with a silicon modulus of 4.0 crumbled and only at a cooling rate of 3 °C/min.

Conclusion

The cooling rate and the chemical composition of slags significantly affect their crumbling. It increases with decreasing cooling rate and as the slag compositions move away from the boundaries of the dicalcium silicate primary crystallisation region. However, a higher silicon modulus results in a lower crumblability.

The granulometric composition of crumbled slags is affected by their chemical composition more than by their cooling rate.

Based on the data obtained, from a technological point of view, the most promising are slags with a silicon modulus not exceeding 2.85 – 3.00, the composition of which is inside or close to the phase triangle bounded by the $12CaO\cdot7Al_2O_3 – 2CaO\cdot SiO_2 – 2CaO\cdot Al_2O_3$ lines.

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