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Effect of boric anhydride on viscosity of slags used in electric melting of metallized siderite concentrate

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Abstract. The Bakal deposit located in the Southern Urals near the city of Bakal, Chelyabinsk region, is one of the largest deposits of carbonate iron ores (siderites). The total deposit of siderites is about 1 billion tons. They are not in demand among metallurgists because of their low iron content and high magnesium content. At the same time, the Urals metallurgical enterprises are suffering from shortage of iron ore raw materials including steelmaking ore raw materials. The high purity of siderites in terms of phosphorus and non-ferrous metals makes it possible to use methods of coke-free metallurgy for their processing. Pyrometallurgical processing of siderites including their reduction roasting in a rotary furnace followed by grinding and magnetic separation allows obtaining a concentrate to be used as a steelmaking raw material having metallization degree above 90 % and a waste rock content under 3 – 7 %. Calculations showed that the costs of electricity used for melting scrap metal and metallized siderite concentrate containing 30 % of waste rock and loaded into the furnace at temperatures above 1000 °C are close. We propose a siderite processing method including reduction of the initial ore in a rotary furnace, and melting of resulting metallized concentrate hot loaded (at temperatures above 1000 °C) into a furnace. The empty rock of metallized siderite concentrate contains a large percentage of magnesium oxide that makes it refractory. To obtain liquid slag, it is proposed to add boric anhydride in the form of colemanite. To assess the B_2O_3 effect on melting of the metallized siderite oxide phase in the process of electric melting, studies on the viscosity correlation of the magnesian steelmaking slag containing B_2O_3 with temperature and its composition were carried out. It was found that at the discharge temperature (1600 °C) the resulting magnesia slag with the ratio of MgO/SiO_2 in the initial siderite equaling to 0.75 – 1.25 has a low viscosity (less than 3.65 P).

Keywords: iron ore raw materials, Bakal siderites, ore processing, roasting, metallization, viscosity, slag, colemanite, boron oxide

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Влияние борного ангидрида на вязкость шлаков электроплавки металлизованного сидеритового концентрата

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Аннотация. Бакальское месторождение, расположенное на Южном Урале вблизи города Бакал Челябинской области, является одним из крупнейших месторождений карбонатных железных руд (сидеритов). Общие запасы сидеритов составляют около 1 млрд т. Они не пользуются спросом у металлургов из-за низкого содержания железа и высокого содержания магния. В то же время металлургические предприятия Урала испытывают дефицит железорудного сырья, в том числе сталеплавильного. Высокая чистота сидеритов по фосфору и цветным металлам позволяет использовать для их переработки методы бескоксовой металлургии. Пирометаллургическое обогащение сидеритов, включающее их восстановительный обжиг во вращающейся печи с последующим измельчением и магнитной сепарацией, позволяет получить концентрат со степенью металлизации более 90 % и содержанием пустой породы менее 3 – 7 %, пригодный в качестве сырья для сталеплавильного производства. Расчеты показали, что затраты электроэнергии на плавку металлического лома и металлизованного сидеритового концентрата, содержащего 30 % пустой породы, и загружаемого в печь при температуре выше 1000 °C, близки. Предложен способ переработки сидеритов, включающий восстановление исходной руды во вращающейся печи и плавку получаемого металлизованного концентрата, в горячем виде (при температуре выше 1000 °C) загружаемого в сталеплавильную печь. Пустая порода металлизованного сидеритового концентрата содержит большое количество оксида магния, что делает ее тугоплавкой. Для получения жидкого шлака предложено использовать добавку борного ангидрида в виде колеманита. Для оценки влияния B_2O_3 на плавление оксидной фазы металлизованного сидерита в процессе электроплавки проведены исследования корреляции вязкости магнезиального сталеплавильного шлака, содержащего B_2O_3, с температурой и его составом. Обнаружено, что при температуре выпуска (1600 °C) образующийся магнезиальный шлак обладает низкой вязкостью (менее 3,65 Пз) при соотношении MgO/SiO_2 в исходном сидерите, равном 0,75 – 1,25.

Ключевые слова: железорудное сырье, бакальские сидериты, обогащение руды, обжиг, металлизация, вязкость, шлак, колеманит, оксид бора

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Introduction

Carbonate (siderite) iron ore deposits span across various regions worldwide, including Austria, Bulgaria, Great Britain, Germany, Poland (Europe); China, Russia, Japan (Asia); Algeria (Africa); USA, Canada, Colombia (America), and numerous other countries [1 – 10]. Presently, the sole metallurgical process employing siderite ores is blast furnace smelting. Before loading into the furnace, siderites undergo enrichment, utilizing various methods depending on the ore's composition: gravity, flotation, magnetic separation, electrostatic separation, and roasting-magnetic techniques. One of the largest known deposits of siderite ore globally is the Bakal deposit, situated near the city of Bakal in the Chelyabinsk Oblast, located in the Southern Urals. The total reserves of siderites in this deposit are estimated to be around 1 billion tons [11; 12]. However, due to their low quality – characterized by low iron content and high magnesium oxide content – siderites are in minimal demand among blast furnace metallurgists. Extraction of the ore falls significantly short of the potential yield based on mining and geological conditions. This shortage exacerbates the scarcity of raw materials faced by the metallurgical industry in the Urals, particularly for steelmaking.

Bakal siderites stand out due to their manganese content, reaching up to 2 %, low phosphorus content (less than 0.02 %), and the absence of non-ferrous metals like copper and zinc. These characteristics render them valuable raw materials for the production of high-quality steels using coke-free metallurgy methods [13; 14].

The methods utilized for direct iron production rely heavily on the quality of the iron ore raw materials employed. The recovery process of high-grade concentrates containing a minimum of 70 % iron is executed in various units such as shaft furnaces, retorts, among others. This procedure aims to achieve a metallization level surpassing 90 % using a specialized gas mixture [15]. For the treatment of low-grade ores, prevalent methods involve their metallization through solid reducing agents in rotary kilns, subsequently followed by the separation of waste rock via grinding and magnetic separation [16]. Extensive research indicates that through the pyrometallurgical enrichment of siderite ores [11; 12], a concentrate boasting over 90 % metallization can be attained. This concentrate, with a waste rock content ranging from 3 to 7 %, proves suitable for steelmaking purposes [17]. In order to facilitate the reduction process at temperatures between 1300 and 1350 °C, the easily fusible waste rock, predominantly composed of quartz-clay shale [19], necessitates prior removal. This removal can be achieved through gravity concentration in heavy suspensions [10], polygradient magnetic separation, or the *X*-ray radiometric method.

The comparison of energy intensity between smelting scrap metal in an electric furnace and smelting metalized siderite concentrate, heated to 1000 °C, containing approximately 30 % waste rock, revealed similar energy consumption per ton of iron in both scenarios. This similarity suggests the feasibility of a smelting technology for metalized siderite concentrate, acquired through pyrometallurgical enrichment in a rotary furnace, without the intermediary stages of grinding and magnetic separation. However, it's crucial to consider that the high magnesium oxide content in the oxide phase of the concentrate will result in the formation of slag during the melting process with a considerably high melting point. This particular characteristic of the slag formation renders the proposed technology inefficient.

The addition of boric anhydride (B_2O_3) to slag is recognized for its capacity to lower the melting temperature of the slag [20]. To assess the impact of B_2O_3 on the melting behavior of the oxide phase of metalized siderite during the electric melting process, research focused on examining the correlation between the viscosity of magnesia steelmaking slag – incorporating B_2O_3 – and its temperature alongside composition.

Table 1

Chemical composition of the initial mixtures

Таблица 1. **Химический состав исходных смесей**

Mixture No.	Content, %						
	SiO_2/MgO	FeO	SiO_2	Al_2O_3	CaO	MgO	MnO
1	0.50	10.0	24.0	4.0	7.0	48.0	7.0
2	0.75	10.0	30.9	4.0	7.0	41.1	7.0
3	1.00	10.0	36.0	4.0	7.0	36.0	7.0
4	1.25	10.0	40.0	4.0	7.0	32.0	7.0

Materials and methods

A vibrating viscometer, utilizing damped oscillations and computerized data processing, was employed for the viscosity study [21].

The proportion of various oxides (iron, calcium, aluminum, manganese) in siderites shows slight variation depending on their location. However, the ratio between silicon oxide and magnesium oxide can fluctuate from 0.5 to 1.25 [11; 12]. Consequently, to examine the viscosity of pure oxides, initial mixtures were formulated. These mixtures replicated the composition of the oxide phase found in a metallized siderite concentrate with a metallization degree of 95 %. The compositions maintained a constant proportion of most oxides while adjusting the SiO_2/MgO ratio within the range of 0.5 to 1.25 (Table 1). These mixtures were supplemented with a material – previously molten and crushed – with a composition closely resembling calcined colemanite. This material contained 8 % SiO_2, 34 % CaO, 4 % MgO and 54 % B_2O_3. It was added at ratios equivalent to 10, 15, and 20 % of its fraction in the charge. This corresponds to 60, 90 and 120 kg of raw colemanite (L.O.I. 30 %) per ton of metallized siderite concentrate. The chemical composition of the examined slags is detailed in Table 2.

The research involved forming tablets from mixtures mirroring the slag compositions under investigation. These tablets were placed in molybdenum crucibles, heated in a resistance electric furnace up to 1600 °C, and subsequently, the viscosity was measured.

The analysis revealed that slags *1 – 3*, featuring a SiO_2/MgO ratio of 0.5, exhibited heterogeneity at temperatures below 1600 °C. In the other slags, distinct regions of high and low-temperature viscosity were observed. Within the high-temperature region, viscosity remained below 3.65 P. As the temperature decreased, viscosity showed a slight increase. However, in the low-temperature region, the change in viscosity was more pronounced. An elevation in the SiO_2/MgO ratio and the addition of colemanite to the mixture led to an increase in B_2O_3 content in the slag and consequently lowered the temperature at which viscosity transitioned to the high-temperature region. The results of these measurements are depicted in Fig. 1.

To analyze the slag viscosities corresponding to the transition from the low-temperature to high-temperature regions, and the respective transition temperatures, experimental design methods were employed [22]. An orthogonal design 2^3 was used, with the SiO_2/MgO ratio as the first factor and the colemanite fraction in the charge as the second factor. The experimental plan

Table 2

Chemical composition of the studied slags

Таблица 2. **Химический состав исследуемых шлаков**

Slag No.	SiO_2/ MgO	Fraction of colemanite, %	Content, %						
			FeO	SiO_2	Al_2O_3	CaO	MgO	MnO	B_2O_3
1	0.50	10.0	9.1	22.5	3.7	9.5	44.0	6.4	4.9
2	0.50	15.0	8.7	21.9	3.5	10.5	42.3	6.1	7.0
3	0.50	20.0	8.3	21.3	3.4	11.5	40.7	5.8	8.9
4	0.75	10.0	9.1	28.8	3.7	9.5	37.7	6.4	4.9
5	0.75	15.0	8.7	27.9	3.5	10.5	36.3	6.1	7.0
6	0.75	20.0	8.3	27.1	3.4	11.5	34.9	5.8	8.9
7	1.00	10.0	9.1	33.5	3.7	9.5	33.1	6.4	4.9
8	1.00	15.0	8.7	32.3	3.5	10.5	31.8	6.1	7.0
9	1.00	20.0	8.3	31.3	3.4	11.5	30.7	5.8	8.9
10	1.25	10.0	9.1	37.1	3.7	9.5	29.5	6.4	4.9
11	1.25	15.0	8.7	35.8	3.5	10.5	28.3	6.1	7.0
12	1.25	20.0	8.3	34.7	3.4	11.5	27.3	5.8	8.9

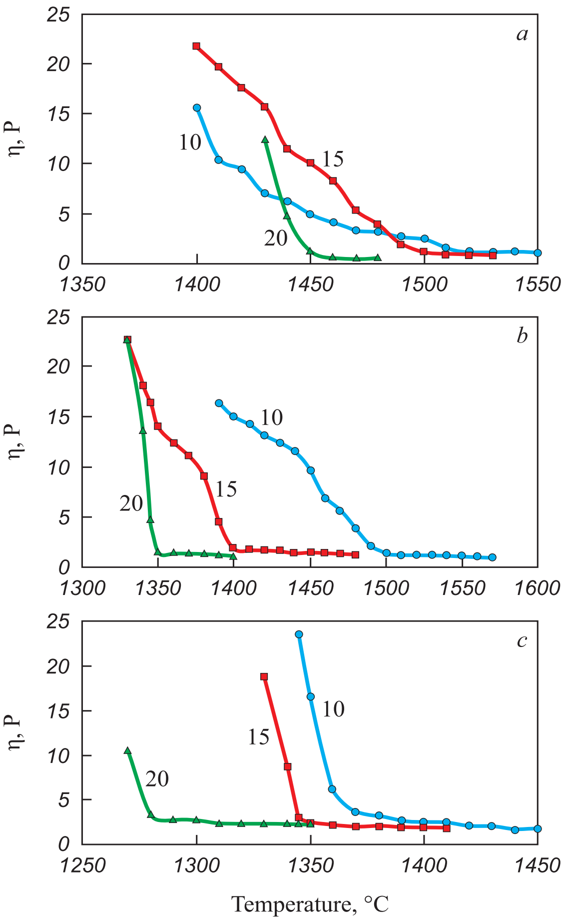

Fig. 1. Change in viscosity of steelmaking slag depending on temperature (numbers near curves – percentage of colemanite) at SiO_2/MgO: 0.75 (*a*); 1.00 (*b*); 1.25 (*c*)

Рис. 1 Изменение вязкости сталеплавильного шлака в зависимости от температуры (цифры у кривых – процент колеманита) при SiO_2/MgO: 0,75 (*a*); 1,00 (*b*); 1,25 (*c*)

Table 3

Plan of the experiment and its results

Таблица 3. **План проведения эксперимента и его результаты**

Test No.	SiO_2/MgO	Fraction of colemanite, wt. %	Temperature, °C	Viscosity, P
1	0.75	10	1520	1.64
2	0.75	15	1500	1.24
3	0.75	20	1450	1.30
4	1.00	10	1500	1.43
5	1.00	15	1400	1.85
6	1.00	20	1350	1.50
7	1.25	10	1370	3.23
8	1.25	15	1345	2.24
9	1.25	20	1280	3.65
10	1.00	15	1400	1.85

and its outcomes are detailed in Table 3 and presented graphically in Figs. 2 – 4.

Fig. 2 provides an overview of the response function depicting slag viscosity and the temperature at which the desired viscosity is achieved, in relation to the SiO_2/MgO ratio and colemanite content.

Fig. 3 shows isolines representing equivalent slag viscosities and the respective temperatures required to achieve those viscosities, contingent on the SiO_2/MgO ratio and colemanite content.

The analysis depicted in Figs. 2 and 3 elucidates the profound impact of the SiO_2/MgO ratio and colemanite content on viscosity. Point *A* in Fig. 3 marks the minimum slag viscosity, attained with a SiO_2/MgO ratio of 0.78 and a colemanite content of 17 %. At a consistent SiO_2/MgO ratio, an increase in colemanite content results in a decrease in the temperature needed to achieve the desired viscosity. For instance, the tem-

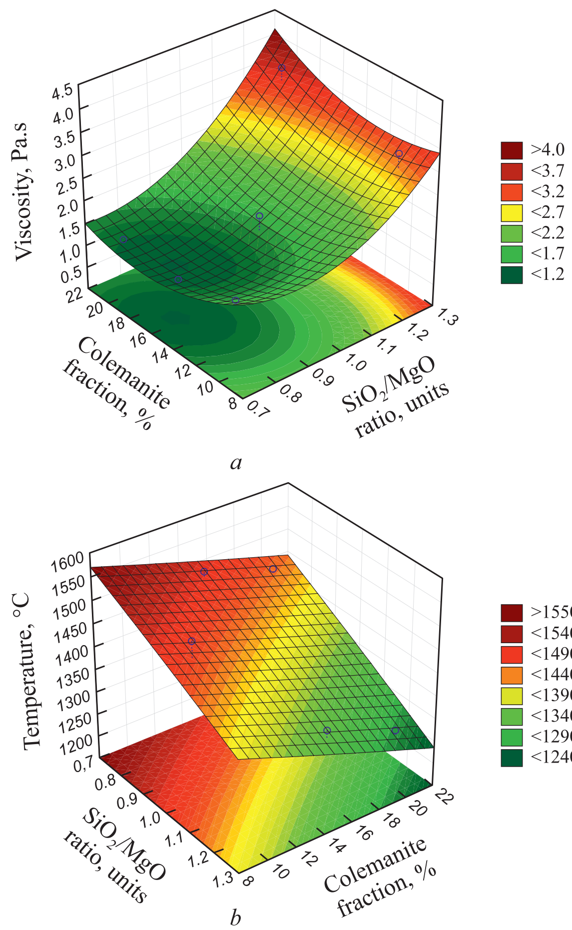

Fig. 2. General view of slag viscosity response function (*a*) and equal temperature at which the required viscosity is achieved (*b*), depending on SiO_2/MgO ratio and proportion of colemanite in the charge

Рис. 2. Общий вид функции отклика вязкости шлака (*a*) и равной температуры, при которой достигается требуемая вязкость (*b*), в зависимости от соотношения SiO_2/MgO и доли колеманита в шихте

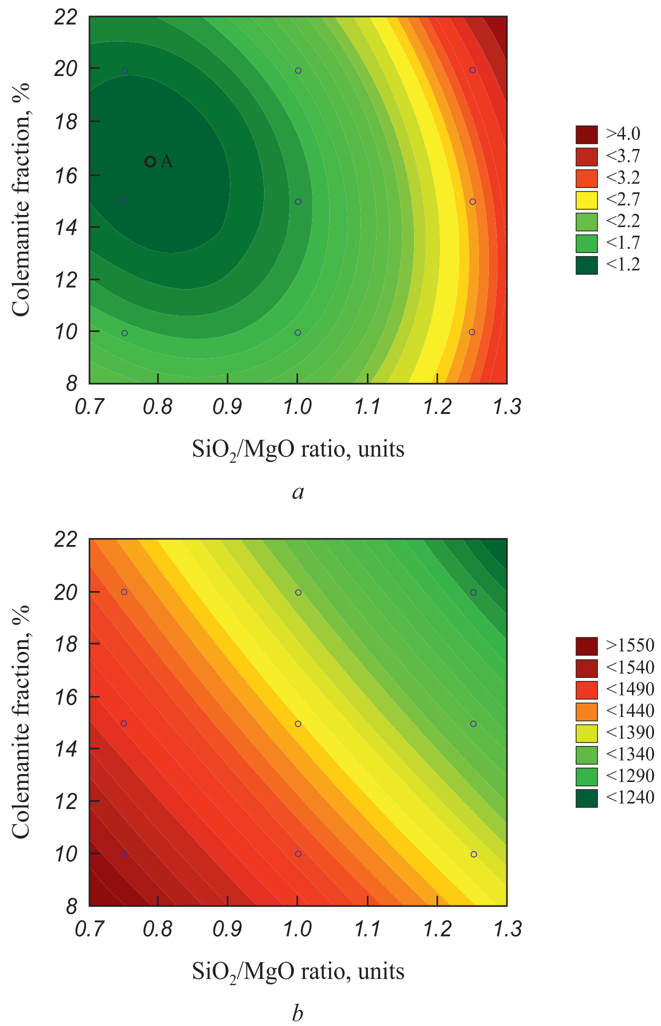

Fig. 3. Isolines of equal slag viscosity (*a*) and equal temperature at which the required viscosity is achieved (*b*), depending on SiO_2/MgO ratio and proportion of colemanite in the charge

Рис. 3. Изолинии равной вязкости шлака (*а*) и равной температуры, при которой достигается требуемая вязкость (*b*), в зависимости от соотношения SiO_2/MgO и доли колеманита в шихте

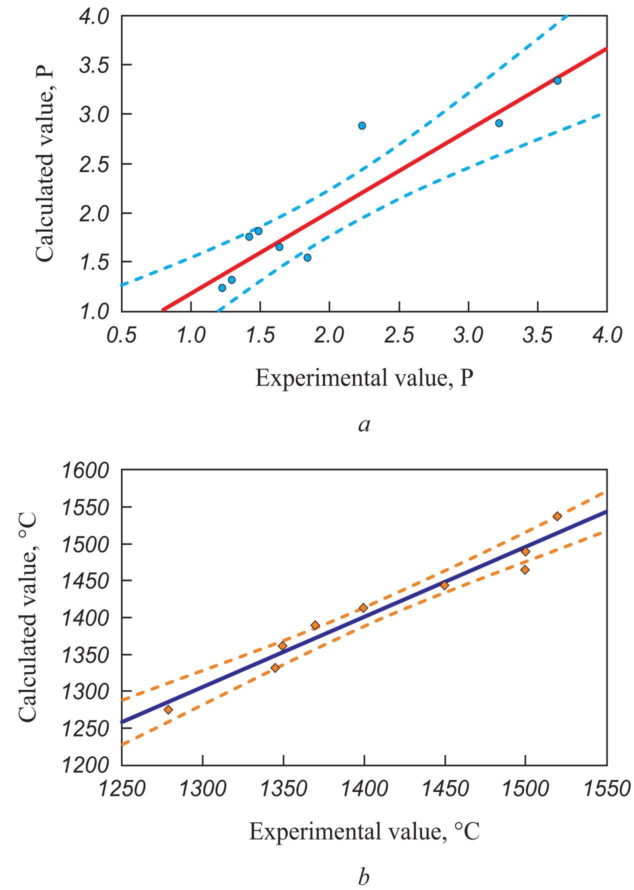

Fig. 4. Correlation diagram of experimental and calculated values of viscosity (*a*) and temperature (*b*)

Рис. 4. Диаграмма корреляции экспериментальных и расчетных значений вязкости (*а*) и температуры (*b*)

perature required to achieve the minimum viscosity with a SiO_2/MgO ratio of 0.78 and a 17 % colemanite content is 1460 °C.

Using the STATISTICA software [24], the experimental results were processed, enabling the calculation of regression equations describing the behavior of the response function (viscosity (*1*) and temperature (*2*)) based on the main factors:

$$\eta = 10.93 - 15.58x + 8.3x^2 - 0.4y + 0.009y^2 + 0.15xy; \quad (1)$$

$$T = 1801.7 - 199.5x - 28.6x^2 - 7.19y + 0.03y^2 - 4.0xy, \quad (2)$$

where x is the SiO_2/MgO ratio, units; y is the colemanite content, wt. %.

Fig. 4 illustrates the comparison between experimental and calculated values of viscosity and temperature to evaluate the model's adequacy determined by the regression equation. The results, assessed via correlation of experimental and calculated data, are visually represented in this figure. The data analysis demonstrates that the regression equation effectively describes the experimental findings. Most of the experimental data points fall within the confidence interval demarcated by the reliability ellipse.

The findings confirm that when the SiO_2/MgO ratio stands at 0.5, the inclusion of colemanite does not yield favorable outcomes. Below temperatures of 1600 °C, the slag retains heterogeneity, impeding the smelting of metallized siderite in electric furnaces. Conversely, in slags featuring a SiO_2/MgO ratio surpassing 0.75, a high-temperature region with viscosity measuring less than 3.65 P is evident. This corresponds to temperatures exceeding 1520 °C, with colemanite fractions ranging from 10 to 20 %. The transition temperature from high to low-temperature regions can be managed by altering both the SiO_2/MgO ratio and the colemanite fraction. Specifically, achieving the same transition temperature involves concurrently increasing the SiO_2/MgO ratio while decreasing the colemanite fraction.

Conclusions

Lump siderite concentrate, reduced to a metallization degree of 95 % in a rotary kiln and loaded into an electric furnace at hot temperatures (above 1000 °C), supplemented with raw colemanite additives within the range of 60 – 120 kg per ton of concentrate, can undergo melting. This process yields a metal-semi-product at outlet temperatures around 1600 °C, suitable for subsequent steel production. Simultaneously, it results in the formation of a homogeneous magnesia slag exhibiting low viscosity (below 3.65 P). This occurs specifically when the original siderite boasts an MgO/SiO_2 ratio ranging from 0.75 to 1.25.

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