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Thermophysical properties of synthetic slags of the FeO – MnO – CaO – Al_2O_3 — SiO_2 system

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ABSTRACT
In the electrometallurgy of manganese alloys, viscosity has an important effect on the production indicators from the physicochemical properties of slag. During the smelting of manganese alloys, the main amount of heat spent on reduction reactions is released due to the current passing through the liquid slag phase, since the resistance of the latter, depending on the composition, affects the completeness of the reduction of manganese from melts. In the case of refined ferromanganese by the silicothermic method, the vast majority of silicon of silicomanganese is refined with higher manganese oxides or it is burned out due to oxygen in the air. The paper presents the results of thermophysical properties of slags representing the FeO - MnO - CaO - Al_2O_3 - SiO_2 system. Laboratory experiments were carried out on synthetic slags by varying the slag basicity CaO / SiO_2 = 1.5 - 1.9 and the concentration of Al_2O_3 = 5 - 15%. As a result, graphs of the dependence of these properties on temperature values are constructed, the activation energies of the viscous flow are calculated and the phase compositions of the experimental slags are determined using a mathematical model of the diagram of the FeO - MnO - CaO - Al_2O_3 - SiO_2 system. Thus, an increase in the concentration of aluminum oxide against its usual level, changing the basicity of the slag, significantly affected the physicochemical properties of the slag and thereby contributes to the adjustment of the parameters of the electric melting process. At the same time, it is advisable to study the interaction of some Physical chemical properties of slags on the melting conditions of manganese ferroalloys in relation to the composition of slags.

Keywords: viscosity, slags of manganese alloys, crystallization temperature, activation energy, phase composition.

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Introduction

In metallurgy, the final slags of manganese alloys, namely for refined grades of ferromanganese, represent the FeO – MnO – CaO – Al_2O_3 - SiO_2 system. The reduction processes during the smelting of refined grades of ferromanganese pass through the slag phase, so the study of the viscous properties of slags is obvious. As is known, the viscosity of the slag in the desired limit should contribute to the rapid runoff of metal droplets into the alloy, the rapid mutual diffusion of oxides and reducing agent, and consequently, the rapid flow of the process, and, finally, allow optimal distribution of the temperature gradient in the melt [1].

In some cases, the viscosity of the slag determines the performance of metallurgical units, such as the interaction of slag with refractory materials, and metal losses with slag. Viscosity is one of the main parameters characterizing the physical properties of oxide melts. The viscosity of a liquid is related to its structure and is determined by interparticle interaction, therefore, its study, along with other physicochemical properties, allows us to evaluate the structure of metal and slag melts, the nature, and magnitude of the interaction between their components.

The thermophysical properties of synthetic slags of the FeO – MnO – CaO – Al_2O_3 – SiO_2 system were investigated.

Experimental part

To study the physical properties of slags (viscosity and temperature of the beginning of crystallization), pre-prepared synthetic mixtures were used, since metal remains and foreign impurities are present in industrial slags.

Slag samples for the study were prepared from reagents of the brand purely for analysis by fusing mixtures in a graphite crucible in a Tamman resistance furnace.

Synthetic materials were calcined to a constant weight at 800 – 850 C before fusion. The suspension of powdered synthetic materials was thoroughly mixed beforehand and then melted in a furnace. After fusion, the resulting slags were submitted for chemical analysis. As a result, the composition of the resulting slags was close to the industrial slags of the process of obtaining manganese alloys (Table 1).

The studied slag samples were characterized by a ratio of CaO/SiO_2 = 1.5; 1.7; 1.9 and different content of Al_2O_3. Such dynamics of composition variation made it possible to trace the influence of alumina and magnesium oxide on the viscosity and crystallization temperature of slags.

The viscosity of the slag was measured with an electro vibration viscometer in molybdenum crucibles with a current of purified argon. The installation diagram is shown in Figure 1.

The high sensitivity of the vibrating viscometer is due to the fact that it operates on resonant vibrations, and the viscosity of the melt violates the resonance conditions [[2], [3], [4]]. To determine the thermophysical properties of the slags of the FeO – MnO – CaO – Al_2O_3 - SiO_2 system, a pre-crushed slag weight (15-20 g) was immersed in a molybdenum crucible with an inner and outer diameter of 17 mm and 30 mm, respectively, and a height of 60 mm. After melting the slag sample (1550-1650 ° C), the contents in the crucible were mixed with molybdenum rods to average the composition. In the center of the crucible, a molybdenum spindle with a diameter of 2 mm and a length of 40 mm was introduced to a depth of 10 mm from the melt surface using a screw lift, then cooling measurements were made at a speed of 3 degrees/min until complete crystallization. Then the hardened slag was melted again to extract the spindle of the viscometer and remove the slag from the crucible by freezing on a molybdenum wire. The temperature in the furnace was measured with a tungsten-rhenium thermocouple TR 5/20, the hot junction of which in a corundum cover was fed through a recess to the bottom of the crucible. The viscosity was recorded by the value of the electromotive force (mV) on a digital millivoltmeter.

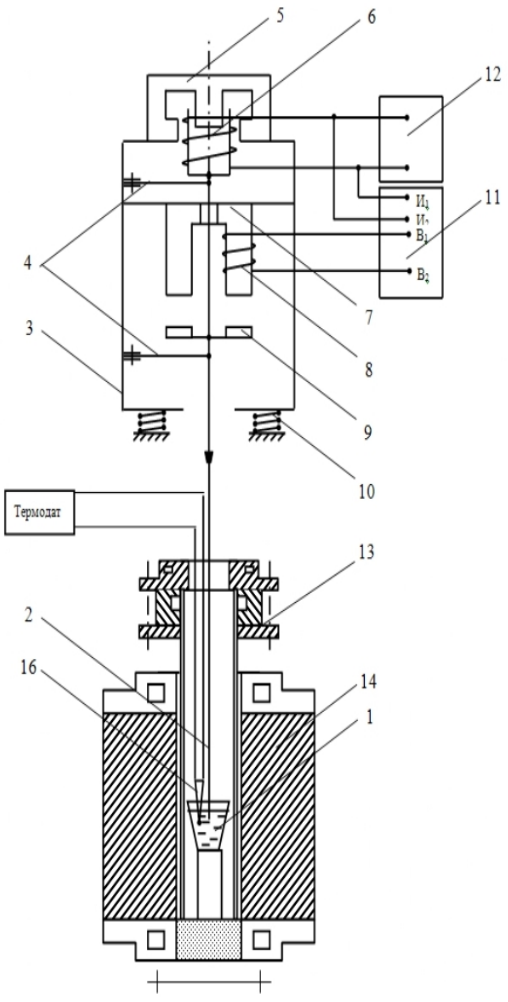


Figure 1 - Diagram of an electro vibration viscometer

1 - crucible; 2 - vibrating rod; 3 - viscometer housing; 4 - flat springs; 5 - ring magnet; 6 - measuring coil; 7 - vibrator core; 8 - vibrator coil; 9 - vibrator armature; 10 - shock-absorbing springs; 11 - auto generator; 12 - digital voltmeters; 13 – lid; 14 - Tamman furnace; 15 - inert gas supply tube; 16 – thermocouple

Table 1 - Chemical composition of the slag

№	Composition, %											
	Chemical						Phase					
	FeO	MnO	CaO	SiO_2	Al_2O_3	CaO/SiO_2	F	M	M_2S	C_2AS	C_2S	CA
1	8.9	19.65	39.01	25.92	6.52	1.51	8.90	7.79	16.86	17.53	48.92	-
2	4.86	16.07	40.35	26.89	11.83	1.50	4..86	3.62	17.70	31.80	42.00	-
3	2.68	15.48	39.32	26.2	16.32	1.50	2.68	3.36	17.24	43.87	32.84	-
4	8.65	15.68	44.42	26.19	5.06	1.70	8.65	10.05	8.00	13.6	59.69	-
5	4.28	14.72	44.66	26.42	9.94	1.69	4.28	8.85	8.34	26.72	51.82	-
6	3.91	15.22	42.45	24.96	13.46	1.70	3.91	10.00	7.42	36.18	42.48	-
7	5.56	15.28	47.12	24.79	7.25	1.90	5.56	15.28	15.22	61.45	-	2.48
8	3.36	13.66	46.45	24.43	12.12	1.90	3.36	13.65	28.33	52.17	-	2.48
9	1.77	14.36	44.01	23.12	16.74	1.90	1.77	14.36	40.77	40.60	-	2.49

Discussions results

Experimental data on the viscosity of slags, the compositions of which are given in Table 1, are shown in Figure 2. The effect of Al_2O_3 concentration on the viscosity of slag with basicity of 1.5; 1.7; 1.9 is shown here.

According to experimental data, the crystallization temperature of the slags was also calculated. It can be determined based on the known exponential dependence of viscosity on temperature [[5], [6]]:

$$\eta = A \cdot e^{-\frac{E}{RT}} \tag{1}$$

where η - the viscosity coefficient; E - the activation energy; T - the absolute temperature; R - the universal gas constant; A - the pre-exponential multiplier. Logarithmic form of expression (1):

$$ln\eta = ln\, A + \frac{E}{RT} \cdot \frac{1}{T} \tag{2}$$

where *lnη* - the segment cut off on the ordinate axis; *E/R* - the angular coefficient of the straight line.

Figure 3 shows the dependence of the logarithm of viscosity (lnη) on the inverse temperature (1/T). As can be seen, an increase in the CaO/SiO_2 ratio in slags affects an increase in the crystallization temperature.

As practice shows, for a homogeneous structural state of slag, the graphical dependence in the coordinate's *ln η - 1/T* must be rectilinear at a certain constant value of E_η. The deviation from the straight line of the dependence of *lnη* from *1/T* indicates the appearance of structural changes in the melt during its cooling. With the development of the crystallization process, a fracture is observed on the graph in the coordinate's *lnη - 1/T*, followed by an increase in viscosity. With the development of the crystallization process, a fracture is observed on the graph in the coordinate's *lnη - 1/T*, followed by an increase in viscosity.

The temperature corresponding to the fracture point determines the crystallization temperature of the studied oxide melt. It can be found from the graph or by solving the equations of the lines together before and after the fracture. In general, they can be written as follows:

$y_a = c + ax$ – for a straight line to a fracture (3)

$y_b = c_1+a_1 \cdot x$ – for a straight line after a fracture (4)

where ln is denoted by «y» lnη, 1/T is denoted by «x», and «c» and «a» represent the free term (lnA) and the angular coefficient (E/R) of equation (2).

The coordinates of the intersection point of two straight lines are as follows (Figure 3):

$$x = -\frac{c_1 - c}{a - a_1} \tag{5}$$

$$y = \frac{c \cdot a_1 - c_1 \cdot a}{a - a_1} \tag{6}$$

Table 2 shows the above-mentioned equations calculated by processing experimental data using the least-squares method, showing the crystallization temperatures ($t_{cr.}$˚C) and the activation energies of the viscous flow of the studied slags (T).

Increasing the concentration of aluminum oxide reduces the viscosity and crystallization temperature of slags in the entire range of the studied basicity. However, the nature of the influence and the absolute values of the decrease in these values, depending on the basicity of the slags, are different, and this is primarily due to the processes of phase formation.

Table 2 - Crystallization temperature and activation energy of viscous flow

CaO/SiO₂	Slags	Equations	$T_{KR.}$,˚C	E, kJ/mol
1.5	1	$\ln_a = -200.97+36.271/T$ $\ln_b = -109.30+19.709/T$	1534	3015.68 1638.67
	2	$\ln_a = -106.86+19.05/T$ $\ln_b = -83.13+14.807/T$	1515	1583.87 1231.10
	3	$\ln_a = -192.67+33.39/T$ $\ln_b = -123.73+21.39/T$	1468	2776.14 1778.43
1.7	4	$\ln_a = -112.38+20.311$ $\ln_b = -105.91+19.107$	1588	1688.72 1588.61
	5	$\ln_a = -114.22+20.023$ $\ln_b = -95.422+16.644$	1524	1664.77 1383.83
	6	$\ln_a = -123.41+21.162$ $\ln_b = -111.13+19.018$	1473	1759.47 1581.21
1.9	7	$\ln_a = -101.03+18.593$ $\ln_b = -73.306+13.373$	1610	1545.88 1111,87
	8	$\ln_a = -161.74+28.987$ $\ln_b = -105.12+18.562$	1568	2410,07 1543.30
	9	$\ln_a = -70.124+12.341$ $\ln_b = -35.36+6.117$	1517	1026.07 508.59

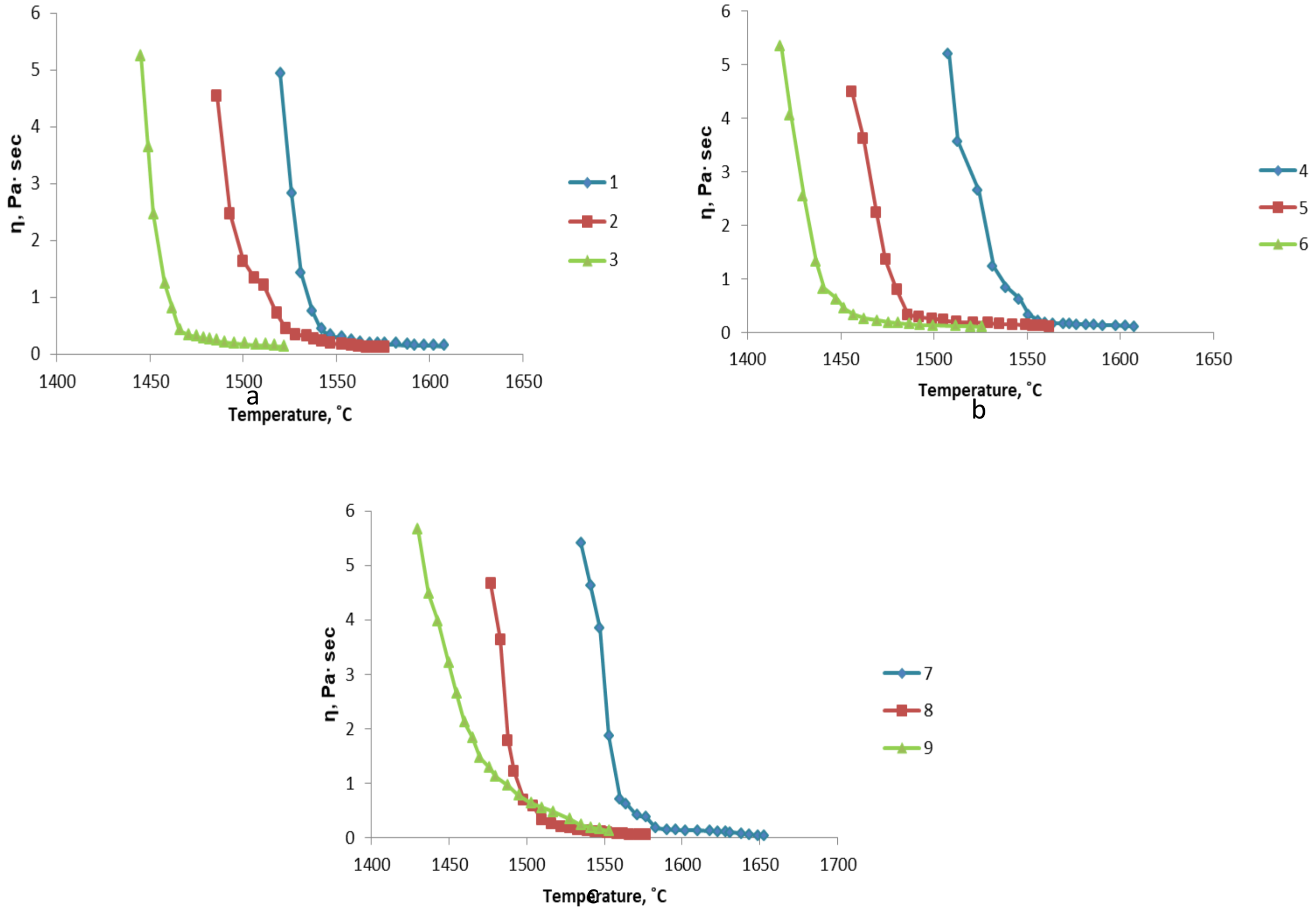
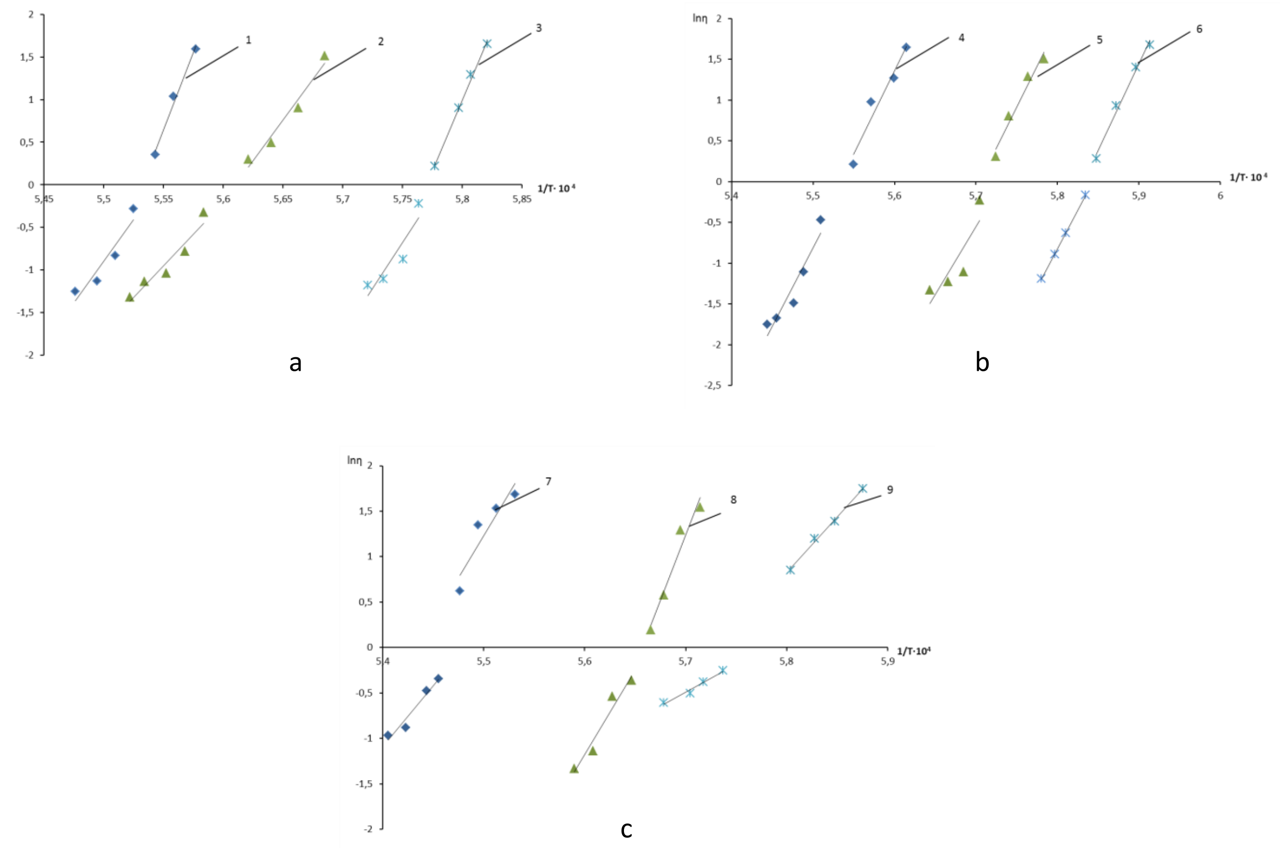


Figure 2 - Dependence of slag viscosity on the temperature at basicities, a - 1.5; b - 1.7; c - 1.9 (figures in the picture of slag numbers according to Table 1)



a

b

c

Figure 3 - Dependence of the logarithm of viscosity on the inverse temperature

The phase composition of the experimental slags was estimated using a mathematical model of the diagram of the FeO - MnO - CaO - Al_2O_3 - SiO_2 system [[7], [8]].

It was found that the slags presented in Table 1 numbered 1-6 have the following phase composition: iron oxide - FeO (indicated in the table as F), manganese oxide - MnO (M), tephroite - $2MnO \cdot SiO_2$ (M_2S), gelenite - $2CaO \cdot Al_2O_3 \cdot SiO_2$ (C_2AS), bicalcium silicate - 2CaO·SiO2 (C_2S) and their normative composition is described by the following expressions:

$F = +1 \cdot F$,
$M = -2{,}367 \cdot S + 1{,}268 \cdot C + 1 \cdot M$,
$M_2S = +3{,}367 \cdot S - 1{,}805 \cdot C$,
$C_2AS = +2{,}688 \cdot A$,
$C_2S = -1{,}688 \cdot A + 1{,}536 \cdot C$.

The calculation shows that with an increase in the basicity of slags due to the CaO additive, there is a decrease in the content of tephroite in them. As a result of the reaction, free manganese oxide and bicalcium silicate are formed [[9], [10]]:

$$2MnO \cdot SiO_2 + 2CaO = 2MnO + 2CaO \cdot SiO_2$$

When the basicity increases to 1.9, the slag composition moves to the pentatope F-M-C_2AS-C_2S-CA, i.e. tephroite is replaced by calcium aluminate (T=1604 °C) [11]. To calculate the phase composition of slags in this pentatope, it is necessary to use the following equations:

$F = +1 \cdot F$,
$M = +1 \cdot M$,
$C_2AS = +9{,}172 \cdot S + 2{,}694 \cdot A - 4{,}917C$,
$C_2S = -2{,}890 \cdot S - 1{,}694 \cdot A + 3{,}085 \cdot C$,
$CA = -5{,}282 \cdot S + 2{,}831 \cdot C$.

Conclusions

By tracking the dynamics of changes in the phase composition, it can be seen that an increase in basicity contributes to the release of MnO in free form from tephroite and thereby contributes to improving the conditions for the reduction of manganese. It is also possible to note an increase in the concentration of Al_2O_3 in the melt, where the temperature range of the beginning of crystallization of the studied slags expands, forming aluminosilicate compounds ($Ca_2Al_2SiO_7$ and $CaAl_2O_4$), the melting point of which is 1550 ° C and

1604 ° C, respectively. At the same time, in order to form a high fluidity of the ore-calcareous melt, it is necessary to ensure a ratio CaO/SiO_2 = 1.5 - 1.7 in the slag and an Al_2O_3 content of at least 10%. Further, with an increase in the CaO/SiO_2 ratio of more than 1.7, the content of the refractory phase (larnite – $2CaO \cdot SiO_2$) having a temperature of 2130 °C will increase, thereby worsening the slag regime of the smelting process of refined ferromanganese. With an increase in the basicity and content of alumina, aluminum atoms are rearranged from gear coordination into anionic complexes of the $(Al_2SiO_7^{4-})_9$ type.

Thus, alumina as a slag-forming component does not worsen the performance of the technology of smelting manganese ferroalloys, in particular for the production of refined ferromanganese. For the technology of smelting refined ferromanganese, the slag region turned out to be optimal in viscosity: M_2S-C_2S-C_2AS with a ratio of CaO/SiO_2 = 1.5 - 1.7. In any other region, properties very undesirable for smelting ferromanganese are the manifested-a high melting point of slags. This contributes to the intensive reduction of silicon from tephroite and may cause an increase in the silicon content in the resulting ferromanganese.

By doing so, we can conclude that it is necessary to correct the slag regime of current production towards high-alumina slags, which allow for higher technical and economic indicators of the production of refined ferromanganese.

Al_2O_3 additives can be produced using alumina-containing materials (bauxite agglomerates) as part of charge materials or aluminum together with siliceous alloys used in metallothermy. However, as practice shows, the latter option is unacceptable due to the high cost and high carbon monoxide of free aluminum.

In this case, the most optimal option for smelting refined grades of ferromanganese is the use of a complex aluminum-manganese-silicon alloy (AlSiMn) in the form of a reducing agent containing up to 15% aluminum along with silicon (50%). With such an aluminum content in the reducing agent, the amount of Al_2O_3 in the slag will fluctuate between 10 -15%.

Conflict of interests. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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FeO – MnO – CaO – Al_2O_3 — SiO_2 жүйесіндегі синтетикалық қождың термофизикалық қасиеті

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ТҮЙІНДЕМЕ

Марганец қорытпаларының электрометаллургиясында қождың физика-химиялық қасиеттерінен тұтқырлықтың өндіріс көрсеткіштеріне әсері маңызды. Марганец қорытпаларын балқыту кезінде жылудың негізгі мөлшері тотықсыздану реакцияларына жұмсалады, ол сұйық шлак фазасынан өтетін ток есебінен жүреді, өйткені соңғысының кедергісі қож құрамына байланысты марганецтің балқымалардан толық тотықсыздануына әсер етеді. Тазартылған ферромарганецті силикотермиялық әдісімен балқыту жағдайында силикомарганец кремнийінің басым бөлігі марганецтің жоғары тотықтарымен тазартылады немесе ауаның оттегі есебінен күйіп кетеді. Жұмыста FeO – MnO – CaO – Al_2O_3 – SiO_2 жүйесі негізіндегі қождардың термофизикалық қасиеттерінің нәтижелері келтірілген. Зертханалық тәжірибелер синтетикалық қожды қолдану арқылы қож негізділігі CaO/SiO_2 = 1,5 - 1,9 және қождағы Al_2O_3 концентрациясы 5-15% аралығында жүргізілді. Нәтижесінде көрсетілген қасиеттердің температуралық шамаларға тәуелділік графигі салынды, тұтқыр ағынның активтену энергиясы есептелді және FeO – MnO – CaO – Al_2O_3 – SiO_2 жүйесіндегі диаграммасының математикалық моделін қолдана отырып, тәжірибелік қождардың фазалық құрамы анықталды. Осылайша, қождың негізділігін өзгерте отырып, алюминий оксидінің әдеттегі деңгейінен концентрациясының жоғарлауы арқылы қождардың физика-химиялық қасиеттеріне айтарлықтай әсер етті, бұл өз кезегінде электр балқыту процесінің параметрлерін түзетуге ықпал етеді. Сонымен қатар, қождардың кейбір физикалық химиялық қасиеттерінің марганец ферроқорытпаларын балқыту жағдайларына әсер етуі қождардың құрамымен байланысты зерттелуі қажет.

Түйін сөздер: тұтқырлық, марганец қорытпаларының қождары, кристалдану температурасы, активтендіру энергиясы, фазалық құрамы.

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Термофизические свойства синтетических шлаков системы FeO – MnO – CaO – Al_2O_3 –- SiO_2

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АННОТАЦИЯ

При электрометаллургии марганцевых сплавов на показатели производства из физико-химических свойств шлака важное значение оказывает вязкость. Поскольку при выплавке марганцевых сплавов основное количество тепла, затрачивается на восстановительные реакции, выделяется за счет тока, проходящего через жидкую шлаковую фазу, поскольку сопротивление последней в зависимости от состава влияет на полноту восстановления марганца из расплавов. В случае рафинированного ферромарганца силикотермическим способом подавляющая часть кремния силикомарганца рафинируется высшим оксидами марганца или же выгорает за счет кислорода воздуха. В работе приведены результаты термофизических свойств шлаков представляющую систему FeO – MnO – CaO – Al_2O_3 — SiO_2. Лабораторные опыты проводились на синтетических шлаках, путем варьирования основности шлака CaO/SiO_2 = 1,5-1,9 и концентрации Al_2O_3 в шлаке в пределах 5-15%. В результате построены графики зависимости указанных свойств от температурных величин, рассчитаны энергии активации вязкого течения и определены фазовые составы опытных шлаков с использованием математической модели диаграммы системы FeO – MnO – CaO – Al_2O_3 – SiO_2. Таким образом, повышение концентрации оксида алюминия против его обычного уровня, меняя основность шлака, существенно повлияло на физико-химические свойства шлаков, и тем самым способствует корректировки параметров процесса электроплавки. При этом взаимодействие некоторых физико-химических свойств шлаков на условия плавки марганцевых ферросплавов целесообразно изучать во взаимосвязи с составом шлаков.

Ключевые слова: вязкость, шлаки марганцевых сплавов, температура кристаллизации, энергия активации, фазовый состав.

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