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Features of mineral formation in the structure of iron ore materials from the position of the state diagram of the system $CaO–Fe_2O_3–SiO_2$

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Abstract. This paper presents the results of a study of the strengthening of iron ore raw materials obtained by oxidative roasting of granules and pellets using gaseous fuel and agglomeration with combustion of solid fuel in the agglomeration layer. Differences in the mechanisms of mineral formation of granules, pellets and agglomerates appear at the stage of liquid-phase strengthening and are due to the different role of iron in forming the strengthening melt. At the same time, in the agglomerate, granules and pellets, iron is in different valence states, affecting the processes' features. Iron can be in a trivalent state in the iron-silicate melt of granules and pellets and is not a silicate-forming component of the charge. The silicate compositions of the binders in the entire studied range of basicities (0.3-1.5) are located along the line of the $CaO–SiO_2$ connection, which is determined using the phase diagram of the $CaO–Fe_2O_3–SiO_2$ system. During agglomeration, the silicon-containing melt is formed under conditions of excess FeO, which directs the process of mineral formation during the creation of the iron-silicate binder of the agglomerate. Under standard agglomeration conditions, silicate binders with a basicity of 1.0-1.5 are formed in the olivine field of the $CaO–FeO–SiO_2$ phase diagram, covering a wide range of compositions. The processes of mineral formation in batches, by hardened methods, both during the firing of granules, pellets, and during agglomeration, have shown that changing the oxidation potential of the gas phase is an effective lever on the path not only to improving the properties of ferrous sand - a waste product of alumina production, but also to creating both new binders and new types of iron ore raw materials suitable for smelting ferrosilicon.

Keywords: *ferrous sand, agglomeration, sintering, iron ore agglomerate, fluxes.*

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1. Introduction

Alumina is known to be produced from bauxite using the Bayer sintering method [1]. These methods produce a large amount of sludge. One of these sludges is called waste (red) sludge. According to sources [2, 3], 0.9 to 1.5 tons of red mud are produced from bauxite for every ton of alumina. To date, red mud has no further use and is stored in sludge fields. The latter occupy vast territories and cause enormous harm to the environment. Such sludge fields occupy 10 to 50 hectares and store millions of tons of waste [4].

Some sources report that between 2.0 billion [5] and 4.0 billion tons of alumina production waste [6] have accumulated worldwide, and vast amounts of money are spent on maintaining such sludge storage facilities. According to [7] alone, the Ural alumina refineries in Russia have accumulated 100-300 million tons of waste.

Alumina production sludge contains a high amount of alkali and therefore poses a danger to the environment. The disaster in Hungary in 2010 (Kolontar) is well known. As a result of a dam break, about 700 thousand tons of sludge were released. People and wildlife suffered, and houses were destroyed [8].

To date, various works on red mud processing have been presented worldwide. However, not all studies conducted are of interest from the metallurgical engineering perspective. Many red mud processing technologies are distinguished by the high cost of implementing the presented studies, and most of them are accompanied by complex and multi-stage processing. Red mud, by its composition, can be used in various areas of industry. The work [9] presents multiple options for using red mud in different sectors. The presented studies consider pyrometallurgical, hydrometallurgical methods, direct use of red mud, and catalysts in the chemical industry. For example, in [10], the processing of red mud is approached critically. In [11], it is proposed to use red mud as a filter for water purification. In [12], it is proposed to subject red mud to pyrometallurgical processing and use it as a building material.

At JSC Aluminum of Kazakhstan, about one million tons of waste sludge accumulates yearly in waste dumps. A significant part of the waste sludge is considered substandard and used only in highway construction as bulk material. As is known, the bauxite deposits of Kazakhstan are distinguished by a large amount of iron and silicon. Therefore, ferrous sands with a

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Fe_2O_3 content of 50-65% are separated at the first stage of bauxite processing by the leaching method. These ferrous sands are sent according to the general scheme to sludge fields and do not find further use. As a result, together with the remains of red and gray sludge, ferrous sands lose their value as iron ore material, mixing with other sludges.

Regarding iron oxide, approximately 10 million tons are already irretrievably lost. Let's consider the iron content in ferrous sands, which are sent to the sludge field in approximately 500 thousand tons per year, as substandard material. This situation is only in Kazakhstan. And according to [13], over 2.7 billion tons of bauxite waste have accumulated worldwide, increasing by 120 million tons annually. However, due to their physical and chemical properties, ferrous sands are suitable for inclusion in metallurgical processing. From this material, it is possible to obtain granules, pellets and agglomerates for use in producing ferrosilicon and other alloys (cast iron and steel). For example, in ferrosilicon production, the obtained granules are quite suitable as a substitute for iron shavings.

When using waste for smelting metals and alloys, two problems are solved: including accumulated waste in metallurgical processing in the form of a semi-finished product and reducing the environmental burden. Ferrosilicon can be obtained by reducing the silicon contained in the studied ferruginous sand and the obtained iron ore agglomerate by over 13%. The need to include iron ore agglomerate in the charge is explained by the fact that oxygen compounds of iron at relatively low temperatures can easily interact with SiO_2 to form silicate melts. The theoretical temperature of silicon reduction by carbon according to the reaction $SiO_2 + 2C = Si + 2CO$ is 1669°C, and the eutectic temperature is 1178°C in the $FeO-SiO_2$ system. When using iron ore materials in the charge during the smelting of ferrosilicon, the processes of melting the charge, rather than reduction, are advanced. With the appearance of silicate melts in the bath of the ferroalloy furnace, the specific electrical resistance of the charge sharply decreases, its components stratify, and silicate slag accumulates, which leads to a breakdown in the furnace operation. Therefore, in connection with the process theory, it is necessary to use the appropriate material that meets the requirements of electric smelting. For this, a thorough study of the mineral formation processes occurring during high-temperature firing in the materials under study (granules, pellets and agglomerates) is necessary.

2. Materials and methods

To work with ferrous sands - waste from alumina production, samples were taken at JSC «Aluminium of Kazakhstan» to obtain iron ore agglomerate in laboratory conditions, in which the optimal parameters for sintering ferrous sands were developed [14] (Figure 1).

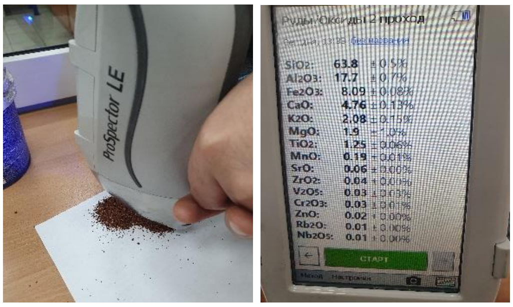
Figure 1. Ferrous sands - waste from alumina production

Also, in the course of the research, a thermodynamic analysis of the phase formation processes during agglomeration was carried out using the state diagrams of the $FeO-CaO-Fe_2O_3-Al_2O_3$ and $FeO-MgO-Fe_2O_3-Al_2O_3$ systems and the effect of dolomite additives in the composition of the sinter batch was studied, which made it possible to determine the optimal amount of magnesium oxide in the batch and identify new phases [15]. The chemical composition of ferrous sands and the resulting iron ore agglomerate are given in Table 1.

Table 1. Chemical composition of the samples studied

№	Materials	Fe_{gen}	Fe_2O_3	FeO	SiO_2	Al_2O_3	CaO	MgO	S	P	ppp
1	Ferrous sand is a waste product of alumina production	31.62	56.78	9.18	6.25	12.16	2.54	1.62	1.12	0.064	12.57
2	Iron ore agglomerate (Basicity 1.2)	55.6	31.33	28.9	13.28	6.58	16.5	3.62	0.86	0.13	3.18

The chemical composition of ferruginous sands was determined by X-ray fluorescence analysis on a portable Prospector 2 LE X-ray fluorescence spectrometer (Figure 2) with a measurement range of chemical elements from Mg to U in the range up to 0.01 %.



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Руды/Оксиды 2 прохода
SiO2:    63.8  ± 0.5%
Al2O3:   17.7  ± 0.7%
Fe2O3:   8.09  ± 0.08%
CaO:     4.76  ± 0.13%
K2O:     2.08  ± 0.15%
MgO:     1.9   ± 1.0%
TiO2:    1.25  ± 0.06%
MnO:     0.19  ± 0.01%
SrO:     0.06  ± 0.00%
ZrO2:    0.04  ± 0.00%
V2O5:    0.03  ± 0.03%
Cr2O3:   0.03  ± 0.01%
ZnO:     0.02  ± 0.00%
Rb2O:    0.01  ± 0.00%
Nb2O5:   0.01  ± 0.00%
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Figure 2. X-ray fluorescence analysis of ferruginous sands

Figure 3 shows samples of iron ore pellets and agglomerate obtained from ferrous sand and waste from alumina production. Optical microscopy in reflected light using a metallographic inverted microscope, METAM LV-32, was used to study the morphology of the agglomerate samples (Figure 4).

(a) (b)

Figure 3. Iron ore pellets (a), iron ore agglomerate (b)

Figure 4. Metallographic inverted microscope METAM LV-32

The micrographs reveal structural features and surface texture characteristic of the sintered phases present in the samples.

3. Results and discussion

In industrial conditions, the main processes of strengthening iron ore raw materials are oxidative roasting granules and pellets using gaseous fuel and agglomeration with solid fuel combustion in the agglomeration layer. Under real roasting conditions, fluxed pellets and agglomerates undergo a similar path of phase transformations at the stage of solid-phase sintering until the appearance of a liquid phase. This path includes dehydration, dissociation and amorphization of minerals of host rocks, as well as decomposition of fluxes with the formation of ferrites.

Differences in the mechanisms of mineral formation of granules, pellets and agglomerates appear at the stage of liquid-phase strengthening and are due to the different role of iron in forming the strengthening melt. In this case, in the agglomerate and pellet, iron is in different valence states, which affects the features of the processes. In the iron-silicate melt of granules and pellets, iron is in a trivalent state and is not a silicate-forming component of the charge. The silicate compositions of the binders in the entire studied range of basicities (0.3-1.5) are located along the CaO–SiO_2 bond line, which is visible in the phase diagram of the CaO–Fe_2O_3–SiO_2 system. According to the diagram shown in Figure 5, the compositions of the silicate binders are in the crystallization region of iron-free mineral phases: cristobalite (SiO_2), wollastonite ($CaSiO_3$), rankinite ($Ca_3Si_2O_7$) and dicalcium silicate (Ca_2SiO_4). In real pellet compositions, trivalent iron enters the melt from calcium ferrites, which are formed at the solid-phase sintering stage and are the primary source of the strengthening melt.

In the zone of maximum temperatures, the finely dispersed fraction of hematite contained in ferruginous sand passes into a melt. When the pellets are cooled under the existing technological firing mode conditions, the melt solidifies predominantly in a glassy form, retaining some of the iron in the trivalent state. A batch of pellets was produced to compare the processes of mineral formation in pellets and agglomerates made from ferruginous sand. However, difficulties associated with pelletizing ferruginous sand arose during their production, since, according to the theory of pelletizing [16-18], the most unfavorable effect on the process is exerted by the particle size in the range of 0.1-1.6 mm.

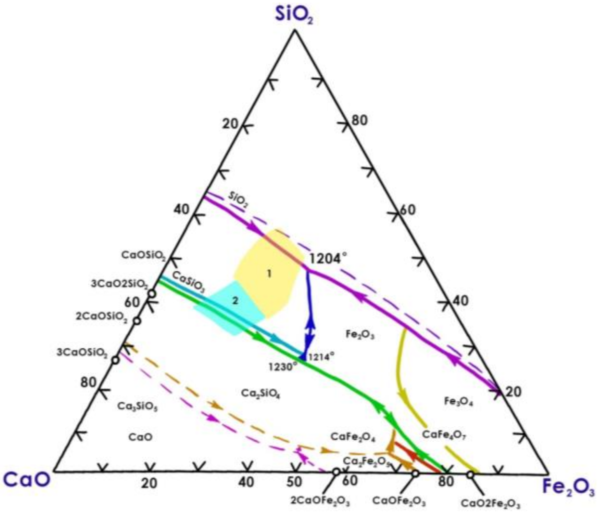


Figure 5. Phase diagram of the CaO - Fe_2O_3 - SiO_2 system. Location of composition areas of silicate binders of fluxed pellets: 1 – compositions providing high strength during reduction of pellets with a SiO_2 content of more than 5%; 2 – the same, with a SiO_2 content of less than 5% [17]

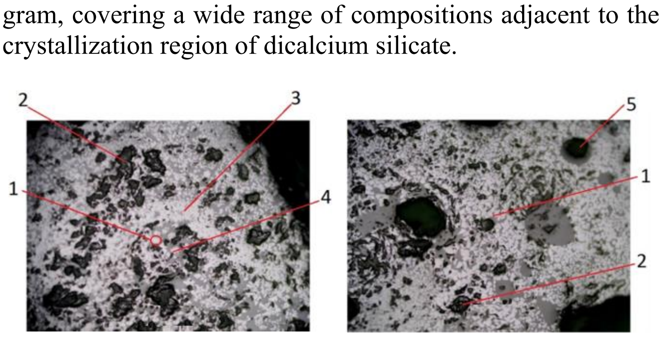
As the data in Table 2 shows, the fractional composition of the studied ferruginous sand corresponds to this range. In this case, the bulk of the ferruginous sand falls on the fraction of 0.2-1.0 mm, making up 63.7% of the total volume.

Table 2. Granulometric composition of ferruginous sands

Fraction, mm	-0.06	-0.2-0.06	-0.5-0.2	-1-0.5	-3-1	-5-3
Units of measurement, %	2.0	14.7	43.2	20.5	6.8	12.8

Finely dispersed iron-containing aspiration dust generated in steelmaking was used to improve the conditions for pelletizing ferruginous sands. The resulting pellets were subjected to firing. During pellet firing at temperatures below the solidus point in air, complete crystallization of the glassy phase occurred with the release of iron in the form of hematite and the formation of iron-free silicate phases. Their composition depended on the basicity of the pellets and corresponded to the crystallization region in the CaO–SiO_2 binary system.

During agglomeration, the silicon-containing melt is formed under conditions of excess FeO, which directs the process of mineral formation during the creation of the iron-silicate bond of the agglomerate. Under standard agglomeration conditions, silicate bonds with a basicity of 1.0-1.5 are formed in the olivine field of the CaO–FeO–SiO_2 phase diagram, covering a wide range of compositions adjacent to the crystallization region of dicalcium silicate.



(a) (b)

Figure 6. General view of the microstructure of the agglomerate (a, b) x100: 1 – magnetite; 2 – wustite; 3 – magnesia; 4 – hercynite; 5 – pores

Based on Figure 6, it can be noted that the content of nonmetallic impurities in magnetite of one genetic variety of iron ore agglomerate remained relatively stable within a specific volume of the sintering batch, but varied significantly at different levels of the sintering cake. The stability of the composition is explained by the fact that only those components of the batch that are within a specific elementary volume are involved in the process of solid solution formation. At the same time, fluctuations in the content of impurities in magnetite of different genetic varieties along the height of the cake are due to the temperature and heat regime of sintering and the amount of liquid phase formed at each level of the sintering batch.

An example is the transformation of grains of the original magnetite into a solid solution of hercynite $FeAl_2O_4$ in magnetite. By analyzing the distribution of zones with different compositions, it is possible to indirectly estimate the state of the ore material in the roasting zone. The composition of the olivine phase in each specific case is determined by the basicity of the melt and the content of divalent iron, which affects its change from calcium-iron olivine, known as ferromonicellite ($CaFeSiO_4$), to ferrous olivine, or fayalite ($FeSiO_4$) [17]. In melts that have been fluxed, the composition does not reach the fayalite phase (Fe_2SiO_4). The maximum content of the fayalite component in the olivine phase can be up to 80%, with 56.5% FeO present in the melt and a minimum melting point of 1130°C, which is confirmed by the data presented in Figure 7. Ferromonticellite and fayalite are two low-temperature phases in the olivine class, with melting points of 1208°C and 1205°C, respectively. Between them, there is a continuous series of solid solutions with a minimum temperature of 1120°C. This corresponds to a composition containing 80% fayalite in a solid solution.

Suppose the melt at the maximum temperatures of the agglomeration process contains high concentrations of divalent iron, which is released as an oxide phase when the agglomerate cools. In that case, its final composition is determined by the oxidation potential of the gas phase.

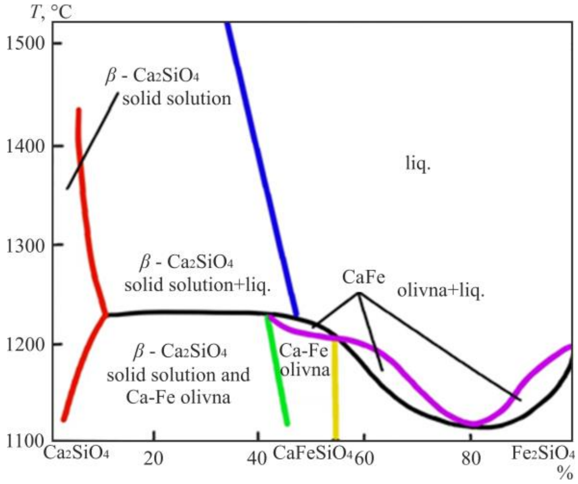


Figure 7. Section of the $CaSiO_4$ - $FeSiO_4$ system [17]

In raw materials of a more complex composition, containing oxides of aluminum contained in ferruginous sands (Table 1) and magnesium, the role of these impurities in forming raw materials is primarily determined by the technological parameters of strengthening [19]. Aluminum oxide, during the firing of pellets, is a component of the binding phases. With different basicity of silicate melts, Al_2O_3 will be part of the ferrite phases or glass. In the composition of silicate binders, Al_2O_3 is an acidic oxide and plays the same role as silica, leading to a decrease in the basicity of the binder. Al_2O_3 is part of the ore, ferrite, and silicate phases during agglomeration. The proportion of aluminum in the composition of various phases at the time of agglomerate formation. The lower the oxidation potential of the gas phase, the more iron oxide and Al_2O_3 in the system in the form of a hercynite solid solution $FeAl_2O_3$ in the magnetite component of the agglomerate. At a high oxidation potential of the gas phase, aluminum remains predominantly in ferrite and silicate phases. The formation of hercynite in a solid solution of magnetite was studied in [18].

4. Conclusions

Magnesium oxide in granules and pellets obtained by oxidative roasting at the stage of liquid-phase strengthening is a source of FeO. Depending on the basicity of the pelletized material, magnesium oxide, together with FeO, is consumed for the construction of silicate phases at a basicity of $CaO/SiO_2 = 0.3\text{-}0.7$. It is part of the magnetite solid solution (Fe, Mg) Fe_2O_4, which is formed at the contact of hematite with a magnesium-containing silicate melt at a basicity of pellets above 0.7. Magnesium oxide in pellets leads to the dissociation of trivalent iron. Magnesium oxide and FeO determine the direction of mineral formation in pellets. Since MgO leads to dissociation of trivalent iron only in the melt, and a small amount of iron dissolves in the pellet melt compared to the agglomerate, the newly formed FeO is not enough to change the mineral formation of the pellet binder with a basicity higher than 0.7. Therefore, all divalent iron and magnesium are spent constructing a magnetite solid solution.

During agglomeration, depending on the ratio of divalent to trivalent iron in the melt, which is determined by the oxidation potential of the gas phase, MgO is part of the ore phase or silicates. The more FeO in the melt, the more MgO in the lattice of the magnetite phase.

Thus, the processes of mineral formation in batches, by hardened methods, both during the roasting of granules and pellets, and during agglomeration, have shown that changing the oxidation potential of the gas phase is an effective lever. In this case, not only are the properties of ferrous sand, a waste product of alumina production, improved, but also new bonds and new types of iron ore raw materials are created in the form of granules, pellets, and agglomerates suitable for use as a charge in smelting ferrosilicon.

Author contributions

Conceptualization: AZ; Data curation: PB, AZ; Formal analysis: AZ-a, AZ, PB; Funding acquisition: AZ; Investigation: AZ; Methodology: OZ, AZ; Project administration: AZ, AZ-a; Resources: OZ, AZ; Software: OZ, AZ; Supervision: AZ; Validation: AZ-a, AZ, PB; Visualization: OZ, AZ; Writing – original draft: AZ-a, AZ; Writing – review & editing: OZ. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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$CaO–Fe_2O_3–SiO_2$ жүйесінің күй диаграммасы тұрғысынан темір кенді материалдарының құрылысындағы минерал түзілуінің ерекшеліктері

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Аңдатпа. Осы жұмыста газ тәрізді отынды пайдалана отырып, түйіршіктер мен шекемтастарды тотығу арқылы күйдіру және агломерациялық қабатта қатты отынды жағу арқылы агломерациялау кезінде алынған темір кені шикізатын беріктендіру зерттеулерінің нәтижелері келтірілген. Түйіршіктер, шекемтастар мен агломераттарда минерал түзілудің механизмдеріндегі айырмашылықтар сұйық фазалық беріктену сатысында байқалады және бұл темірдің беріктендіретін балқыманың түзілуіндегі әртүрлі рөлімен түсіндіріледі. Бұл жағдайда агломератта және түйіршіктер мен шекемтастарда темір әртүрлі валентті күйде болады, бұл процестердің ерекшеліктеріне әсер етеді. Түйіршік пен шекемтастардағы темір-силикатты балқымада темір үш валентті күйде болады және шикізат құрамындағы силикат түзуші компонент болып табылмайды. Барлық зерттелген негізділік ауқымында (0.3-1.5) байланыстырғыштардың силикатты құрамы $CaO–SiO_2$ сызығы бойында орналасады, бұл $CaO–Fe_2O_3–SiO_2$ жүйесінің күй диаграммасын қолдану арқылы анықталады. Агломерация кезінде кремний бар балқыма FeO-ның артық мөлшері жағдайында түзіледі, бұл агломераттағы темір-силикатты байланыстырғыштың минерал түзуші процесін бағыттайды. Агломерацияның стандартты жағдайларында 1.0-1.5 негізділіктегі силикатты байланыстар $CaO–FeO–SiO_2$ жүйесінің күй диаграммасындағы оливиндік өрісте түзіледі және құрамдарының кең ауқымын қамтиды. Түйіршіктер мен шекемтастарды күйдіру мен агломерациялау арқылы беріктендірілген шикізаттарда минерал түзілудің жүруі газ фазасының тотығу потенциалын өзгерту – глинозем өндірісінің қалдығы болып табылатын темірлі құмның қасиеттерін жақсартуда ғана емес, сондай-ақ жаңа байланыстырғыштар мен жаңа түрдегі темір кенді шикізаты жасаудың да тиімді тетігі екенін көрсетті.

Негізгі сөздер: *темірлі құм, агломерация, жентектеу, темір кенді агломерат, флюстер.*

Особенности минералообразования в структуре железорудных материалов с позиции диаграммы состояния системы $CaO–Fe_2O_3–SiO_2$

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Аннотация. В настоящей работе приводятся результаты исследования упрочнения железорудного сырья, полученные при окислительном обжиге окатышей с использованием газообразного топлива и агломерации со сжиганием твёрдого топлива в агломерационном слое. Различия в механизмах минералообразования окатышей и агломерата проявляются на стадии жидкофазного упрочнения и обусловлены различной ролью железа в формировании упрочняющего расплава. При этом в агломерате и окатыше железо находится в разных валентных состояниях, что влияет на особенности процессов. В железосиликатном расплаве окатышей железо находится в трёхвалентном состоянии и не является силикатообразующим компонентом шихты. Силикатные составы связок во всём исследованном диапазоне основностей (0.3-1.5) располагаются вдоль линии соединения $CaO–SiO_2$, которое определяется при использовании диаграммы состояния системы $CaO–Fe_2O_3–SiO_2$. При агломерации кремнийсодержащий расплав формируется в условиях избытка FeO, что направляет процесс минералообразования при создании железосиликатной связки агломерата. В стандартных условиях агломерации силикатные связки с основностью 1.0-1.5 формируются в оливиновом поле диаграммы состояния $CaO–FeO–SiO_2$, охватывая широкий диапазон составов. Процессы минералообразования в шихтах, упрочненными способами, как при обжиге окатышей, так и при агломерации показали, что изменение окислительного потенциала газовой фазы является действенным рычагом на пути не только улучшения свойств железистого песка – отхода глиноземного производства, но и создания, как новых связок, так и новых видов железорудного сырья.

Ключевые слова: *железистый песок, агломерация, спекание, железорудный агломерат, флюсы.*

Publisher’s note