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INVESTIGATION OF PHASE TRANSFORMATIONS DURING THE SINTERING OF FERRUGINOUS SANDS FROM ALUMINA PRODUCTION FROM THE PERSPECTIVE OF MULTICOMPONENT OXIDE SYSTEMS

Aigul Zhunusova ^{1*}, Peter Bykov ¹, Ablay Zhunusov ¹, Anar Kenzhebekova ¹,
Adlet Kulbekov ¹

¹Toraighyrov University, Pavlodar, Kazakhstan

*Corresponding author: Zhunusova Aigul Kairgeldinovna, e-mail: zhunusova.a@mail.ru

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ABSTRACT

This study examines the physicochemical characteristics of ferrous sands generated during alumina production from the perspective of thermodynamic-diagram analysis of multicomponent oxide systems. It was established that the specific properties of these ferrous sands, including their low melting point, fine particle size distribution, and the absence of clay components, hinder their application in conventional sintering processes and subsequent metallurgical treatment. To evaluate the mechanisms of phase formation, the FeO–CaO–Fe₂O₃–Al₂O₃, FeO–MgO–Fe₂O₃–Al₂O₃, and FeO–MgO–CaO–Fe₂O₃ systems, representing the composition of ferrous waste and the resulting slag melts, were investigated. The analysis showed that hercynite (FeAl₂O₄), which adversely affects the reducibility of iron-bearing materials and promotes the formation of low-melting slag phases, may form during the sintering process. The thermodynamic analysis indicates that the introduction of magnesia-containing fluxes can reduce the predicted proportion of hercynite while promoting the formation of more refractory phases, including magnesium and ferrite spinels, thereby providing a theoretical basis for improving the structural stability of the sinter. The thermodynamic analysis confirms the potential of MgO-containing fluxes for the sintering of ferrous sands and demonstrates the necessity of thermodynamic modeling of multicomponent oxide systems for selecting rational burden compositions and producing high-strength sinter with stable physicochemical properties. The results provide a theoretical basis for the development of technologies for the integrated recycling of iron-bearing alumina production wastes and for improving the preparation of iron ore raw materials for subsequent metallurgical processing.

INTRODUCTION

Thermodynamic-diagram analysis (TDA) of multicomponent oxide systems, developed by S.O. Baisanov, is based on the combined consideration of the thermodynamic feasibility of reactions between components or compounds formed within a particular system together with its corresponding phase diagram. When phase equilibrium data for the investigated system are unavailable, the analysis additionally incorporates the general regularities of phase diagrams, isomorphic relationships between complex phases, and the results of mineralogical investigations of the system under consideration (Yessengaliyev et al., 2019; Makhambetov et al., 2021).



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The advantages of thermodynamic–diagram analysis are particularly evident in the investigation of multicomponent systems. This approach makes it possible to construct a phase equilibrium diagram in which each elementary subsystem is thermodynamically independent and may therefore be regarded as a separate quasisystem. For example, during the crystallization of liquid slags, only those compounds (phases) located at the vertices of the corresponding quasisystem, to which the slag composition belongs, will crystallize, whereas the melt will predominantly contain the dissociation products of these compounds. Consequently, a number of important slag properties can be predicted, including the presence of binary, ternary, and higher-order eutectics, as well as variations in liquidus temperature and other physicochemical characteristics. This predictive capability represents one of the principal advantages of thermodynamic–diagram analysis of multicomponent oxide systems (Shabanov et al., 2020; Shabanov et al., 2022; Baisanov et al., 2008).

The application of conventional carbothermic technologies for processing iron-bearing raw materials and industrial wastes is frequently accompanied by the formation of primary and intermediate slags characterized by low melting temperatures and extremely low viscosity. These observations are supported by extensive studies conducted at the Zh. Abishev Chemical and Metallurgical Institute, where phase equilibria in complex oxide systems containing iron oxides and associated components have been investigated. Preliminary studies have demonstrated that the sintering of alumina production ferrous sands results in the formation of hercynite (FeAl_2O_4) (Zhunusova et al., 2024).

Hercynite (FeAl_2O_4) forms a continuous solid solution with magnetite. The occurrence of hercynite-containing solid solutions in magnetite within the sinter is attributed to the preferential crystallization of magnetite from the iron-silicate melt, followed by the incorporation of alumina into its crystal lattice. Magnetite–hercynite solid solutions exhibit extremely poor reducibility, while the presence of aluminum ions significantly increases the reduction initiation temperature. For example, raw materials containing approximately 10 wt.% hercynite in magnetite begin to reduce only at temperatures close to 1000 °C. To clarify the mechanism of hercynite formation, it is necessary to perform a thermodynamic–diagram analysis of the FeO – CaO – Fe_2O_3 – Al_2O_3 , FeO – MgO – Fe_2O_3 – Al_2O_3 , and FeO – MgO – CaO – Fe_2O_3 systems. According to previous studies (El-Hussiny et al., 2011; Zhunusova et al., 2025; Baisanov et al., 2024), during the initial stage of reduction, FeAl_2O_4 decomposes into metallic iron and spinel-type compounds under the action of carbon, whereas at elevated temperatures these spinel phases are transformed into α - Al_2O_3 .

The phase diagrams of the FeO – CaO – Fe_2O_3 – Al_2O_3 , FeO – MgO – Fe_2O_3 – Al_2O_3 , and FeO – MgO – CaO – Fe_2O_3 systems (where Fe represents FeO , F' represents Fe_2O_3 , A represents Al_2O_3 , C represents CaO , and M represents MgO) have previously been extensively investigated and described in detail (Kelamanov et al., 2024; Baisanov et al., 2005; Gabdullin et al., 2024; Baisanov et al., 2022; Tolokonnikova et al., 2022). The first diagram is particularly suitable for describing the phase composition of highly iron-rich industrial wastes and the primary slags produced during the manufacture of pig iron, high-carbon ferromanganese, and low-silicon ferrosilicon. In contrast, the second diagram is more appropriate for interpreting the phase composition of final slags formed during the production of ferrous metals and ferroalloys.

MATERIALS AND METHODS

Ferrous sands generated during alumina production possess several specific characteristics, including a low melting temperature, a fine particle size distribution, and the absence of clay constituents. These characteristics complicate their utilization in conventional sintering technologies and their subsequent use in pig iron and ferrosilicon production. Therefore, a comprehensive understanding of the phase equilibria in the FeO – CaO – Fe_2O_3 – Al_2O_3 , FeO – MgO – Fe_2O_3 – Al_2O_3 , and FeO – MgO – CaO – Fe_2O_3 systems, which adequately represent the composition of these materials during metallurgical processing, is required.

The primary objective of this study was to investigate the mechanisms governing phase transformations in iron oxide-based systems and to determine the optimal chemical compositions of the investigated systems for both the ferrous waste materials and the burden components. Appropriate selection of the compositional regions is expected to promote the formation of high-strength sinter and stable bonding slag melts with favorable physicochemical properties.

To achieve this objective, the most promising approach is the adaptation of the sintering process to the specific characteristics of ferrous sands. This requires thermodynamic and physicochemical modeling of the sintering process based on the analysis of multicomponent oxide systems to determine rational burden compositions and the conditions necessary for producing high-quality sinter.

The investigated ferrous sands obtained from alumina production contain not only iron oxides but also several other valuable oxide components. The chemical composition of the investigated material is presented in Table 1.

Table 1. Chemical composition of ferrous sands from alumina production

Material	Total Fe	SiO ₂	MnO	Al ₂ O ₃	MgO	CaO	S	P	C
Ferrous sands from alumina production	61,0	7,8	-	19,0	0,5	5,7	0,2	-	-

To intensify the sintering process, fluxing materials are introduced into the burden mixture. Lime is the most widely used flux in industrial sintering plants. The addition of lime improves granulation behavior and increases the mechanical strength of the resulting sinter, thereby enhancing bed permeability and increasing sintering productivity. However, if the limestone is insufficiently ground, fine particles may not be completely heated and dissociated during sintering. As a result, the lime formed during limestone decomposition does not fully react with or dissolve in the melt. Unreacted lime particles remain visible in the sinter and significantly reduce its resistance to storage and transportation. Furthermore, upon contact with moisture, lime hydrates to form portlandite, causing degradation of the sinter microstructure. Therefore, magnesia-containing fluxes can be considered more suitable fluxing materials for the agglomeration of ferrous sands.

RESULTS AND DISCUSSION

The use of dolomitized and magnesia-containing fluxes intensifies the sintering process and enhances the mechanical strength of the resulting agglomerate. Magnesium-bearing additives have a beneficial effect on the strength of fluxed sinter. Magnesium carbonates participate in reactions with the components of the sintering burden and can dissolve in iron-containing melts. Magnesium ions subsequently crystallize and are incorporated into the crystal lattice of dicalcium silicate (2CaO·SiO₂), forming a solid solution based on β -Ca₂SiO₄. Even small additions of magnesium inhibit the polymorphic transformation of β -Ca₂SiO₄ by stabilizing the high-temperature modifications of dicalcium silicate. As a result, internal stresses within the iron ore sinter are significantly reduced, thereby improving its structural stability.

According to the microscopic and X-ray diffraction analyses reported by Malysheva et al. (2019), the synthesized products were identified as single-phase materials, confirming the formation of FeAl₂O₄ (hercynite) in the quaternary FeO–CaO–Fe₂O₃–Al₂O₃ system. Based on its softening and melting temperatures, hercynite is classified as a low-melting mineral and exhibits relatively high electrical conductivity. Consequently, smelting of iron ore sinter produced from ferrous sands is expected to be accompanied by the formation of low-melting slags. This behavior

can be explained by the fact that slag formation proceeds more rapidly than iron oxide reduction, resulting in the premature generation of a liquid slag phase. Such a phenomenon may adversely affect the operating conditions and technological parameters of the smelting furnace.

However, this unfavorable effect can be minimized through appropriate process control. If the compositional changes occur in the vicinity of the spinel phase fields, including iron monoaluminate, magnesium spinel, and ferrite spinel, the resulting slags are expected to exhibit significantly higher refractoriness. Therefore, to suppress the formation of undesirable low-melting slags, it is advisable to introduce fluxing materials containing magnesium oxide or calcium oxide during the sintering of ferrous sands.

The application of MgO-fluxed sinter in pig iron production promotes the formation of more refractory final slags compared with conventional low-melting slags. To evaluate the influence of magnesium oxide on the physicochemical properties of the sinter, it is necessary to determine how the phase composition of both the sinter and the slag phase changes with increasing MgO addition and, on this basis, establish the optimum flux dosage.

Thus, from the standpoint of the multicomponent oxide systems $\text{FeO}-\text{CaO}-\text{Fe}_2\text{O}_3-\text{Al}_2\text{O}_3$, $\text{FeO}-\text{MgO}-\text{Fe}_2\text{O}_3-\text{Al}_2\text{O}_3$, and $\text{FeO}-\text{MgO}-\text{CaO}-\text{Fe}_2\text{O}_3$, the effect of introducing MgO-based fluxes into the sintering burden should be investigated through changes in the phase composition of the sinter, since these changes determine its physicochemical properties.

The $\text{FeO}-\text{CaO}-\text{Fe}_2\text{O}_3-\text{Al}_2\text{O}_3$ system (Figure 1) contains a reciprocal subsystem analogous to the magnesia-containing system (Fe^{2+} , $\text{Ca}^{2+}/\text{Fe}_2\text{O}_4^{2-}$, $\text{Al}_2\text{O}_4^{2-}$). This subsystem is characterized by the principal diagonal $\text{F}'\text{A}$ (hercynite) – CF (calcium monoferrite), which is determined by the Gibbs free energy of the following reaction:



In addition, the presence of the stable $\text{F}'-\text{C}_2\text{F}$ boundary, corresponding to the reaction:



modifies the phase relationships of this system, as shown in Figure 1. As a consequence of these two binary subsystems, three quasi-ternary subsystems— $\text{F}'-\text{F}'\text{A}-\text{C}_2\text{F}$, $\text{F}'\text{A}-\text{C}_2\text{F}-\text{CF}$, and $\text{F}'\text{A}-\text{CF}-\text{A}$ —are formed within the tetrahedron. These subsystems separate all calcium aluminates from magnetite ($\text{F}'\text{F}$), as illustrated by the separated parts of the $\text{F}'-\text{C}-\text{F}-\text{A}$ system relative to the reciprocal system Fe^{2+} , $\text{Ca}^{2+}/\text{Fe}_2\text{O}_4^{2-}$, $\text{Al}_2\text{O}_4^{2-}$.

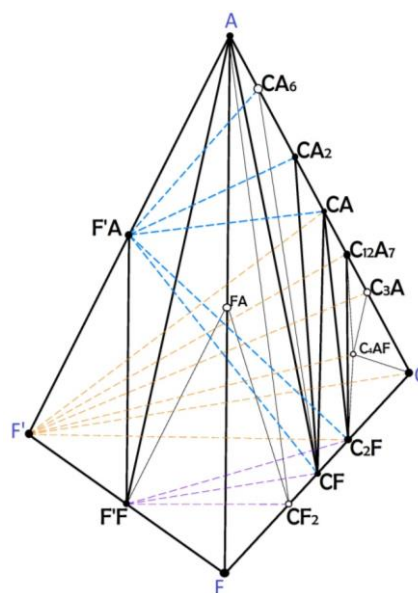


Figure 1. Phase diagram of the $\text{FeO}-\text{CaO}-\text{Fe}_2\text{O}_3-\text{Al}_2\text{O}_3$ system ($\text{F}'-\text{C}-\text{F}-\text{A}$)

According to Zhunussova et al. (2024), reactions between iron-bearing components of the sintering burden and the fluxes predominantly result in the formation of low-melting liquid phases localized within the calcium ferrite phase field. These melts contain magnetite (Fe_3O_4), wüstite (FeO), hercynite (FeAl_2O_4), and calcium monoaluminate ($\text{CaO} \cdot \text{Al}_2\text{O}_3$). These phases are schematically illustrated in Figure 1.

The formation of calcium aluminates as independent phases becomes possible only after the completion of all chemical reactions and the establishment of phase equilibrium. Their occurrence is primarily confined to regions enriched in lime or magnesia-containing compounds.

However, the X-ray diffraction analysis of the ferrous sands presented in Figure 2 did not reveal the presence of calcium monoaluminate among the identified crystalline phases.

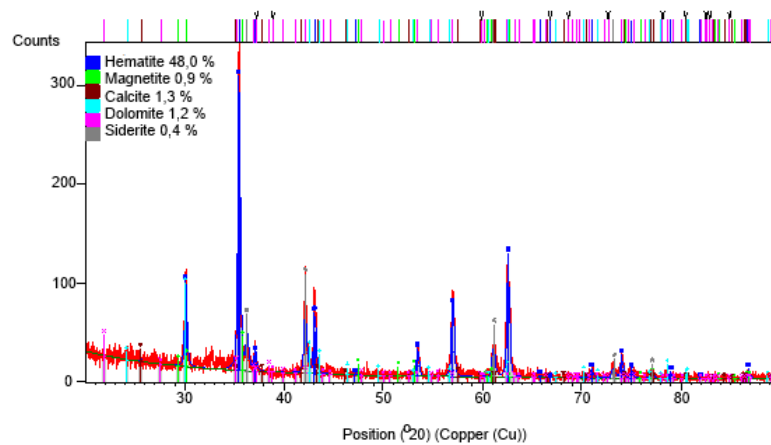


Figure 2. X-ray diffraction pattern of ferrous sands from alumina production

The coexisting phase assemblages listed in Table 2 for each congruently melting phase of the system can be used to identify stable polytopes within the overall six-component $\text{F}'\text{-C-Mg-F-A-S}$ system. As shown in Table 2, the C-Mg-F-A subsystem contains hercynite as one of its stable phases.

In the $\text{FeO-MgO-Fe}_2\text{O}_3\text{-Al}_2\text{O}_3$ system ($\text{F}'\text{-Mg-F-A}$) (Figure 3), the phase relationships are governed by the following reactions:

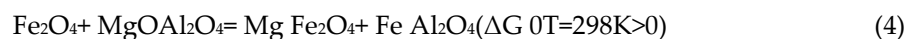


Table 2. Coexisting Phases in the C-Mg-F-A System

Nº	Phase	Abbreviated Formula	Coexisting Phases
1	Wüstite	F'	$\text{F}'\text{F}, \text{C}_2\text{F}, \text{C}, (\text{C}_4\text{AF}), (\text{C}_3\text{A}), \text{C}_{12}\text{A}_7, \text{CA}, \text{F}'\text{A}$
2	Alumina	A	$\text{F}'\text{A}, \text{F}'\text{F}, (\text{FA}), (\text{F}), (\text{CF}_2), \text{CF}, (\text{CA}_6), \text{CA}_2$
3	Hematite	(F)	$\text{F}'\text{F}, (\text{FA}), \text{A}, (\text{CF}_2), \text{CF}$
4	Lime	C	$\text{C}_2\text{F}, (\text{C}_4\text{AF}), (\text{CA}_3), \text{C}_{12}\text{A}_7, \text{F}'$
5	Magnetite	$\text{F}'\text{F}$	$\text{F}', (\text{F}), (\text{FA}), \text{A}, \text{F}'\text{A}, (\text{CF}_2), \text{CF}, \text{C}_2\text{F}$
6	Calcium Monoferrite	CF	$(\text{F}), (\text{C}_2\text{F}), \text{F}'\text{F}, \text{F}'\text{A}, \text{A}, (\text{CA}_6), \text{CA}_2\text{CA}, \text{C}_2\text{F}$
7	Dicalcium Ferrite	C_2F	$\text{CF}, \text{F}'\text{F}, \text{F}', \text{F}'\text{A}, \text{A}, (\text{CA}_6), \text{CA}_2\text{CA}, \text{C}_2\text{F}$
8	Dodecacalcium Heptaaluminate	C_{12}A_7	$\text{CF}, \text{F}'\text{F}, \text{F}', \text{F}'\text{A}, \text{CA}, \text{C}_{12}\text{A}_7, (\text{C}_4\text{AF}), \text{C}$
9	Calcium Monoaluminate	CA	$\text{C}_{12}\text{A}_7, \text{C}_2\text{F}, \text{CF}, \text{F}', \text{F}'\text{A}, \text{CA}_2$
10	Calcium Dialuminate	CA_2	$\text{CA}, \text{CF}, \text{F}'\text{A}, (\text{CA}_6), \text{A}$
11	Hercynite	$\text{F}'\text{A}$	$\text{F}', \text{F}'\text{F}, \text{CF}, \text{C}_2\text{F}, \text{CA}, \text{CA}_2, (\text{CA}_6), \text{A}$

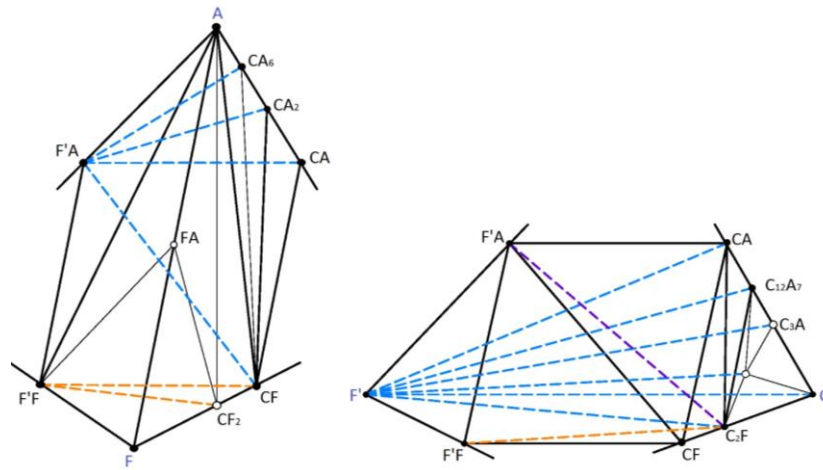


Figure 3. Separated Sections of the F'-C-F-A System

These reactions divide the lateral faces of the tetrahedron into six stable quasi-quaternary subsystems, including the reciprocal subsystem Fe^{2+} , $\text{Mg}^{2+}/\text{Fe}_2\text{O}_4^{2-}$, $\text{Al}_2\text{O}_4^{2-}$, whose principal diagonal is $\text{Fe}_3\text{O}_4\text{-MgAl}_2\text{O}_4$. This reciprocal subsystem is formed as a consequence of Reaction (4) (Table 3).

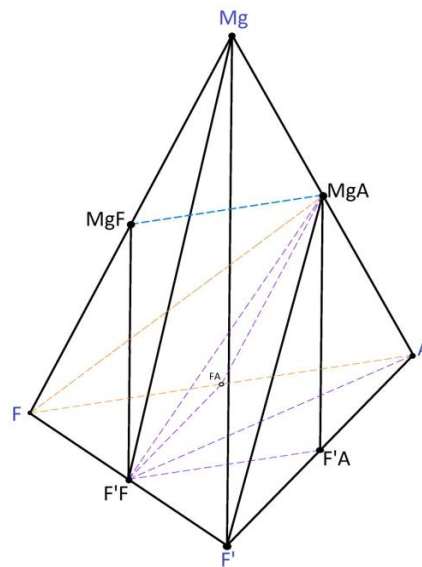


Figure 4. Phase diagram of the $\text{FeO-MgO-Fe}_2\text{O}_3\text{-Al}_2\text{O}_3$ system (F'-Mg-F-A)

As shown in Table 3 and Figure 4, hercynite is also present in the F'-Mg-F-A system. However, the thermodynamic-diagram analysis indicates that the introduction of magnesium oxide into the sintering burden during the agglomeration of ferrous sands can decrease the predicted proportion of hercynite while promoting the formation of additional magnesium-containing phases. In particular, magnesium spinel (MgAl_2O_4) and magnesium ferrite spinel (MgFe_2O_4) become important components of the predicted phase assemblage, contributing to the formation of more refractory phase associations.

The $\text{FeO-MgO-CaO-Fe}_2\text{O}_3$ system (F'-Mg-C-F) (Figure 5) has a relatively simple phase topology [17]. One of its faces (C-F'-Mg) contains no intermediate compounds, whereas the remaining faces contain analogous compounds, namely F'F, CF, and MgF, which form an extensive series of solid solutions within the isomorphous quasi-ternary subsystem F'F-CF-MgF.

Table 3. Coexisting Phases in the F'-Mg-F-A System

Nº	Phase	Abbreviated Formula	Coexisting Phases
1	Wüstite	F'	F'F, F'A, Mg, MgA
2	Alumina	A	F'A, F'F,(FA),(F), MgA
3	Hematite	(F)	F'F,MgF, MgA,(FA), A
4	Magnesia	Mg	MgF, F'F, F', MgA
5	Magnetite	FF	(F), F', MgF, MgA,(FA), A,F'A
6	Iron Aluminate (Hercynite)	F'A	F',F'F, MgA,A
7	Magnesium Spinel	MgA	A, F'A, F', F'F, (F),MgF,Mg, (FA)
8	Magnesium Ferrite Spinel	MgF	(F),F'F,MgA, Mg

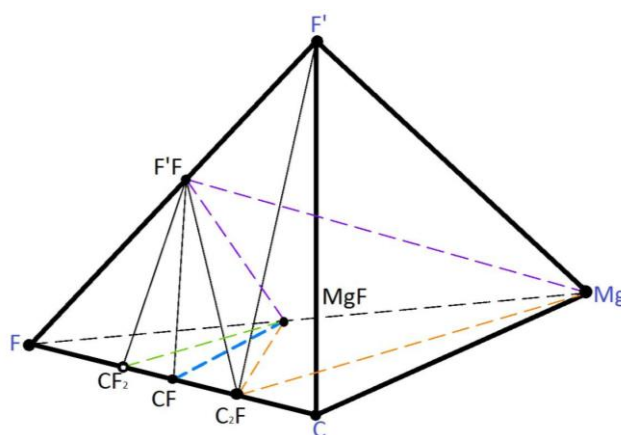


Figure 5. Phase Diagram of the FeO–MgO–CaO–Fe₂O₃ System (F'–Mg–C–F)

The FeO–MgO–CaO–Fe₂O₃ system is characterized by a relatively simple phase topology, consisting of five elementary tetrahedra (Figure 5 and Table 4), considering the modifications introduced into the phase diagrams of the C–Mg–F and F'–C–F subsystems discussed previously.

The phase assemblages presented in Table 4 indicate the presence of hematite, wüstite, and magnetite. However, similarly to the previously considered systems, the introduction of magnesium oxide into the sintering burden through dolomite fines promotes the formation of new phases, namely periclase (MgO) and magnesium ferrite spinel (MgFe_2O_4).

Table 4. Coexisting phases in the F'-Mg-C-F system

Nº	Phase	Abbreviated Formula	Coexisting Phases
1	Hematite	(F)	F'F, (CF ₂), MgF,CF
2	Wüstite	F'	F'F, C ₂ F, C,Mg
3	Magnetite	F'F	(F), (CF ₂), CF, CF ₂ , MgF, Mg,F'
4	Lime	C	Mg, C ₂ F, F'
5	Magnesia	Mg	MgF, F'F, F', MgA
6	Magnesium Ferrite Spinel	MgF	(F), (CF ₂), CF, C ₂ F, Mg, F'F
7	Dicalcium Ferrite	C ₂ F	C,Mg,MgF, F'F, F',CF
8	Calcium Monoferrite	CF	C ₂ F, F'F,MgF, (CF ₂)

CONCLUSIONS

The thermodynamic-diagram analysis of the FeO–CaO–Fe₂O₃–Al₂O₃, FeO–MgO–Fe₂O₃–Al₂O₃, and FeO–MgO–CaO–Fe₂O₃ systems made it possible to identify the principal phase transformations occurring during the sintering of ferrous sands from alumina production.

The analysis demonstrated that hercynite (FeAl_2O_4) is a stable phase in the investigated multicomponent oxide systems and may form during the sintering of ferrous sands containing a high proportion of Al_2O_3 . The formation of hercynite is undesirable because it is associated with low reducibility and can contribute to the formation of low-melting slag phases during subsequent metallurgical processing.

The introduction of MgO-containing fluxes changes the phase relationships in the investigated systems. According to the thermodynamic-diagram analysis, increasing the MgO content promotes the formation of more refractory magnesium-containing phases, including magnesium spinel (MgAl_2O_4) and magnesium ferrite spinel (MgFe_2O_4), while decreasing the relative amount of hercynite in the predicted phase assemblage. The $\text{FeO-MgO-Fe}_2\text{O}_3\text{-Al}_2\text{O}_3$ and $\text{FeO-MgO-CaO-Fe}_2\text{O}_3$ systems therefore provide a theoretical basis for selecting MgO-containing fluxes for the sintering of ferrous sands.

The results indicate that the rational selection of the flux composition should be based on the phase relationships of the multicomponent oxide system and the formation of refractory bonding phases. In particular, MgO-containing fluxes can be considered a promising direction for improving the structural stability and physicochemical characteristics of sinter produced from alumina-production ferrous sands.

CONFLICT OF INTEREST: The authors declare no conflict of interest.

INSTITUTIONAL REVIEW BOARD STATEMENT: Not applicable. This study did not involve human participants, animals, or any procedures requiring approval by an Institutional Review Board or equivalent ethics committee.

INFORMED CONSENT STATEMENT: Not applicable. This study did not involve human participants; therefore, informed consent was not required.

DATA AVAILABILITY STATEMENT: The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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Information about authors



Zhunusova Aigul Kairgeldinovna – Toraighyrov University, Pavlodar, Kazakhstan, PhD, Senior Lecturer, Metallurgy, materials science and mechanical engineering.

E-mail: zhunusova.a@mail.ru

ORCID: <https://orcid.org/0000-0001-8842-6433>



Bykov Petr Olegovich – Innovative University of Eurasia (InEU), Pavlodar, Kazakhstan, First Vice-Rector – Vice-Rector for Academic Affairs, Professor, Candidate of Technical Sciences, Metallurgy, Materials Science and Mechanical Engineering.

E-mail: bykov_petr@mail.ru

ORCID: <https://orcid.org/0000-0001-7540-7882>



Zhunusov Ablay Kairtasovich – Toraighyrov University, Pavlodar, Kazakhstan, Professor and head of the department “Metallurgy”, Metallurgy, metallurgical processes, ferroalloy production, recycling of technogenic waste, agglomeration technologies

E-mail: zhunusov_ab@mail.ru

ORCID: <https://orcid.org/0000-0001-9119-9737>



Kenzhebekova Anar Yerbolatovna – Toraighyrov University, Pavlodar, Kazakhstan, Associate Professor (Docent), Metallurgy, agglomeration processes, industrial waste utilization.

E-mail: kenzhebekova_psu@mail.ru

ORCID: <https://orcid.org/0000-0002-8111-6755>



Kulbekov Adlet Mauletovich – Toraighyrov University, Pavlodar, Kazakhstan, Doctor of Philosophy (PhD) in Chemistry, Materials Science, Chemical Engineering

E-mail: Adlet.kam@mail.ru

ORCID: <https://orcid.org/0009-0001-2059-6214>