

Ferrum extraction in cast iron via reduction smelting of red mud

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High-iron Bayer red mud, including more than 30 % Fe, is considered as poor iron ore. Owing to global iron deficiency during last decades, the problem of efficient use of iron which is containing in red mud with high Fe content attracts most and most attention. This research includes development of the technological route for Fe extraction from red mud in cast iron via reduction smelting with consequent accelerated cooling for metal separation from slag. Influence of different experimental parameters (such as temperature, basicity and reduction time) on Fe extraction from red mud was examined in details. The results displayed that separation between metal and slag was complete. Maximal Fe extraction in cast iron was obtained at the temperature 1450 °C and made in average 88.5 % (when using fluxes without soda) and 91.5 % (with soda addition). Microstructure of the samples was determined, it showed the structure of hyper-eutectic white cast iron (ledeburite + graphite). No non-metallic inclusions were revealed in the structure of samples. Optimal experimental results are very important for achievement of large-scale and high-efficient red mud recovery.

Key words: red mud, cast iron, reduction smelting, ferrum, recycling, alumina production wastes, red mud processing, technological route of recycling.

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Introduction

The Bayer method is the main technology for alumina production at present time, about 90 % of total alumina production worldwide is based on this method due to lower energy consumption and better efficiency in comparison with sintering process. The Bayer process is used in alumina cleaning from bauxite ore and gives solid high-alkali salt-sodium accompanying product which is called bauxite reminder – red mud (RM). It has calculated annual production 150 mln tons, global reserves at present time are evaluated as 4.6 bln tons [1–4]. Increase of valorization of bauxite reminders, which are forming in the Bayer process, is a serious problem for the alumina industry both by economical and ecological causes [4].

Red mud contains essential amount of valuable metals, such as Fe (6.8–71.9 %), Ti (2.5–22.6 %), Al (2.12–28.4 %) and rare earth elements (REE), and can be considered as potential secondary source of raw materials. Its use for extraction of these metals will correspond to the principles of circular economics in mining industry and lowering of the effect on the environment [3, 4]. International aluminium institute (IAI) formulated the aim to use up to 20 % of red mud until 2025 in its road map of alumina technology; but at present time only 2–3 % of annual red mud production is recovered [5].

For many years large efforts were directed on development of sustainable technologies of red mud processing in order to transform RM from wastes to valuable products and secondary source of raw materials, with reducing their storage sites and connected expenses. The most known laboratory and semi-industrial researches in this area in Russia were carried out by the Institute of metallurgy of Ural branch of RAS [6], by the Baikov Institute of metallurgy and material science of RAS and National university of science and technology “MISiS” [2]. The researches on red mud processing were started in the middle of XX century, they are continuing worldwide and at present time hundreds methods of RM recycling are developed and suggested. There are numerous publications about this problem; up to 35,000 articles appeared annually during last 5 years only in Google Academy electronic library. That’s why this article emphasizes on surveys [2, 3, 6, 14].

Storage and burial of these solid wastes, as well as their use in building industry, in manufacture of cement, mineral wool, ceramics etc. remain the basic methods of their recycling [2]. Despite the fact that red mud from different plants are rather different in their chemical and phase composition, Fe-containing minerals and other valuable metals are their main components [2]. Thereby they can be considered first of all as sources of raw materials for metallurgical industry. The extraction methods for valuable metals (such as alumin-

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ium, ferrum, titanium, scandium, rare-earth elements) from red mud are presented by the modifying version of conventional metallurgical technologies. Hydrochemical processing methods are usually based of use of mineral acids – sulfuric, hydrochloric and nitric ones [1, 3, 5, 7]. Multi-component composition of red mud hampers selective extraction of the concrete metal, and large amounts of acids are required in this case for alkali neutralization in mud pulp, as well for transition of the components in a soluble state. Choice of a technological route for extraction depends on relationship between alumina and silica, Fe content and total amount of rare-earth elements [7].

The most of acid and alkali methods consider only alumina extraction technology. However, the extracted tails are not used consequently. To rise efficiency of red mud use, the new method of calcification and carbonization was suggested [8, 9]. This method allows to avoid restriction on aluminium / silicon relationship in alumina production via Bayer technology. Additionally, sodium and aluminium content reduce substantially, providing principally cheap and harmless secondary use of red mud. The research is directed on provision of theoretical ground for industrial application of the method of calcification and carbonization and attracts attention as the new possible method for pure alumina production and red mud processing.

Due to depletion of iron ore raw materials reserves, the researches in the field of Fe extraction from high-iron red mud. Many investigations of Fe extraction from Bayer red mud with high Fe content were conducted using physical, hydro- and pyrometallurgical methods [2, 3, 8-14]. Fe extraction from high-iron red mud has large researching value and importance. The work [3] systematize Fe extraction methods, which are divided by three techniques: physical separation, pyrometallurgical technology (reduction smelt-

ing and reduction roasting) and hydrometallurgical technology (acid leaching) (Fig. 1).

Magnetic separation is one of the main physical methods. Its deficiency is low content of magnetic concentrate and low degree of Fe extraction (55 % and 35 % respectively). The work [15] shows Fe extraction 91 % for the following optimal experimental parameters: red mud / carbon / additive in relation 100:50:4 and roasting at 700 °C during 20 min. However, total Fe content in concentrate during magnetic separation made only about 60 %.

In the process of hydrometallurgical extraction large amount of acid is used; it can not only cause corrosion of equipment, but also will lead to forming of large amount of processed acid and, respectively, to secondary contamination [2, 14]. At present time investigations of Fe pyrometallurgical reduction present the most interest [2].

Efficient Fe reduction from 48.23 % in red mud to 90.12 % in magnetic concentrate via red mud roasting and consequent magnetic separation is displayed in the work [12]. Fe extraction degree makes 94.95 % for the following experimental conditions: roasting at the temperature 1050 °C during 60 min, magnetic field intensity 0.1 T with additives of 6 % Na_2SO_4 and 6 % Na_2CO_3 . The work [9] describes carbothermal reduction roasting of red mud with sodium and potassium carbonates and consequent magnetic separation. Thermodynamic analysis of behaviour of Fe and Fe-free components in the process of carbothermal reduction was carried out using software HSC Chemistry 9.98 and FactSage 7.1. Influence of addition of alkali carbonates as well as roasting duration and temperature on Fe metallization degree, Fe grains size and magnetic separation process was examined experimentally. The following conditions of reduction roasting were recognized as the best ones:

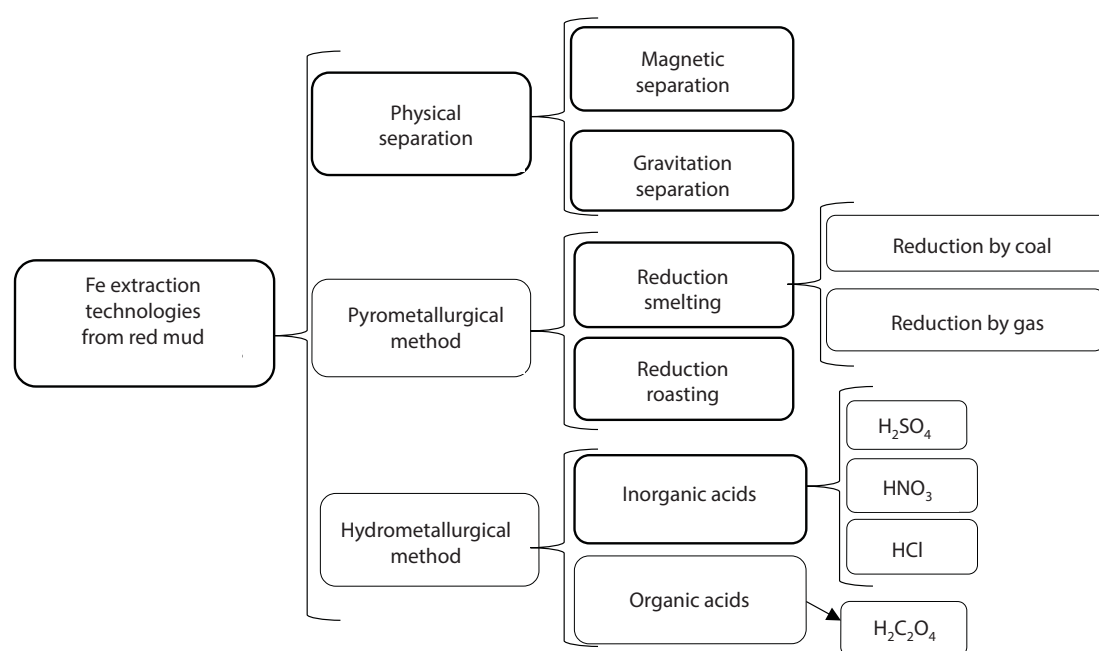


Fig. 1. Classification of the main technologies for Fe extraction from red mud [3]

Content of elements in material, % (mass.)											
Fe	O	Al	Si	Ca	Ti	C	Na	Mn	Cr	S	P
28.15	41.29	8.35	7.12	6.29	2.36	1.20	2.12	0.785	0.05	0.71	0.23
Content of elements in material, % (mass.)											
Cu	Zr	Pb	Zn	Ni	Ga	Cl	Sr	Mg	K	P3M	
0.02	0.17	0.04	0.07	0.06	0.007	0.056	0.09	0.28	0.34	0.22	

Phase	Content, % (mass.)	Name
Fe ₂ O ₃	38.5	Hematite
CaCO ₃	8.16	Calcite
Na ₆ [Al ₆ Si ₆ O ₂₄] · (CaCO ₃) · (H ₂ O) ₂	22.80	Cancrinite
Ca ₃ Al ₂ · (SiO ₂) · (OH); Ca ₃ Al ₂ (SiO ₄) _{3-x} (OH) _{4x} (x = 1.5–3.0)	11.40	Catoite
(Fe, Al) · O · (OH)	4.00	–
AlO(OH)	2.04	Diaspore
CaTiO ₃	1.43	Perovskite
Al ₂ Si ₂ O ₅ · (OH) ₄	18.00	Kaolinite
TiO ₂	1.10	Anatase
TiO ₂	1.96	Rutile

addition of 22.01 % K₂CO₃, processing at the temperature 1250 °C during 180 min.

The work [11] presents cast iron obtaining with Fe extraction 96.52 %, with low Mn and Si content and relatively high S and P content via direct reduction of pellets with coal and red mud. At the same time, the part of reduced Fe and non-reduced Fe in the form of Fe₂SiO₄ remains in slag. The work [13] examined influence of different experimental conditions (temperature, time of smelting and basicity) on total Fe content in cast iron and the coefficient of Fe extraction in the process of reduction smelting. Fe content in metal and Fe extraction coefficient made 94.06–80.16 % with basicity 0.9 for smelting at the temperature 1600 °C during 30 min.

Unfortunately, no one from all previously developed technologies for RM use led to essential commercial application due to their economical inexpedience. The detailed review of the suggested methods and procedures for red mud processing is presented in the works [2, 3, 7, 14]. As a rule, the researchers note that economical calculations were not included in the aims of investigation owing to complete absence of this research to the concrete places of possible putting into practice of the results. Significant difference in prices for raw materials and their transportation and delivery costs enable impossibility of even simple economical calculation. Free-of-charge industrial wastes (or even with prepayment and delivery from wastes producers) instead of expensive natural materials will be always high-profitable from the point of view of reasonable sense [16, 17]. It is possible to achieve the real results using evaluation methods of ecological and economical efficiency for developed technological solutions, such as comparative, structural-logical, economical-statistical analysis and real options [16, 17].

The aim of this research was development of technological route and calculation of material flows for Fe extraction

via reduction smelting of red mud with carbon and in presence of fluxes.

Materials and methods

Red mud from SUAL JSC – affiliate of SUAL Holding, Kamensk-Uralskiy, was used as initial raw material, its average chemical and phase composition is presented in the **Tables 1** and **2**. X-ray photographs for phase identification were registered using X-ray phase and X-ray spectral analyses by X-ray diffraction meter XRD-6000 Shimadzu, using CuK radiation. To provide more precise phase identification, information and searching system RetrieveQQPA and database PDFICDD are used. Capturing of spectra images for X-ray spectral analysis was carried out by automatic wave X-ray fluorescent spectrometer Shimadzu XRF-1800 (with Rh anode).

Material is mainly presented by hematite, cancrinite and kaolinite. Fe with its content about 30 % (mass.) is mainly connected in hematite. Aluminium, calcium, sodium, titanium are presented with content from 1 to 10 %, carbon and silicon from non-metals are included in this range. Aluminium is presented in cancrinite, kaolinite and catoite. Titanium is located in the phases of anatas, rutile and perovskite. Larger oxygen amount in chemical composition is confirmed by oxide phases. Several amorphous phases, which are not determined by X-ray fluorescent analysis, are presented in the material composition. Analyzing red mud is fine-grained material with grain size 4–100 µm; average grain size makes 17 µm.

This works is devoted to research of pyrometallurgical reduction of red mud, which implies preparation of charge material containing of red mud and carbon reducing agent. To delete excessive physical moisture, containing in red mud

samples, RM preliminary drying was carried out during 4 hours at the temperature 100–120 °C. Test charge of dry red mud with mass 100 g was stirred thoroughly with preliminarily dried components of fluxes ($\text{Ca}(\text{OH})_2$, soda, silicon oxide) and coal fines in previously calculated amounts. Flux introduction in charge allows to decrease liquidus temperature and slag viscosity, what improves metal coagulation and separation between metal phase and slag, as well as slag tapping and the process of consequent comminution (slag with soda adding is less hard).

All chemical reactants correspond to KhCh grade. Charge was loaded in a graphite crucible and heated in a smelting furnace up to the temperature of reduction of Fe oxides. Consequent reduction of Fe oxides in charge was carried out by carbon reducing agent with Fe saturation by carbon; then reduced charge was molten with obtaining of metal phase presented by cast iron and slag, and afterwards cast iron was separated from primary slag. Experiments for Fe extraction were carried out within the temperature range 1420–1470 °C, melting time was 60 and 90 min, reduction was conducted with and without soda presence. Chemical composition of obtained metal phase was determined by X-ray spectral analysis.

Obtained Fe-containing material was analyzed using microscope Observer.A1m, Carl Zeiss, magnification x200. Pickling was conducted by pickling agent for special steels.

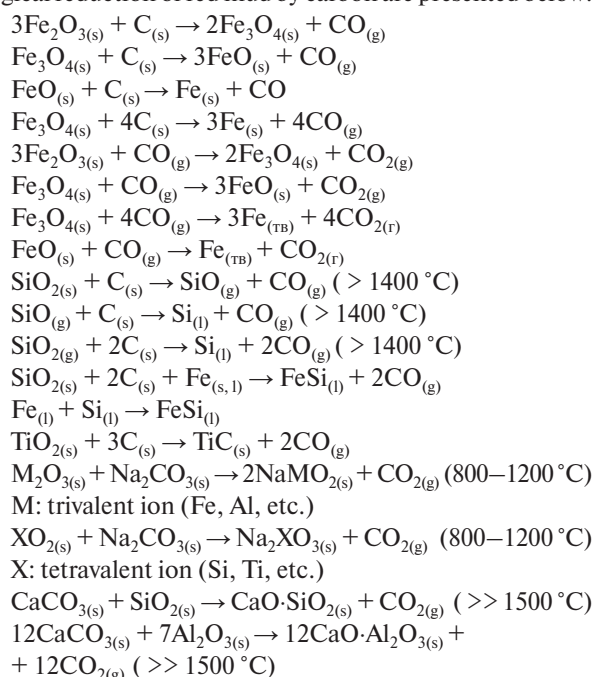
Slags obtained after reduction smelting were preliminarily comminuted in a steel mortar. Chemical and phase composition and behaviour during heating were examined using X-ray spectral and X-ray phase analyses. Additional examination of chemical composition was carried out via the methods of wet chemistry. Slags were subjected to leaching by water, alkali, sulfuric and hydrochloric acids. Preliminary gravimetric separation of slags to heavy and light fractions was conducted in order to decrease consumption of reactants and material flows. However, preliminary concentration didn't provide essential separation of components, according to the data of X-ray structural analysis. Leaching of light fraction of slags was conducted, extraction of valuable

components was obtained significantly lower than that for initial slags. Content of Fe and Al oxides in slag was determined by complexometric method with trilon B in presence of sulfosalicylic acid. Content of aluminium α -oxide was determined by gravimetric method after dissolution of slag test charge in solution of aluminium sulphate and hydrochloric acid with consequent extraction, drying and calcination at the temperature 1100 °C. Carbon amount was determined by gravimetric method after boiling of 5 g of comminuted sample in hydrochloric acid with consequent filtration and calcination of precipitate at the temperature 600 °C. Titanium oxide in slag was determined by photometric method with hydrogen peroxide.

Results and discussion

Theoretical model of the process was determined in the base of analysis of red mud composition and potentially possible reactions. It was aimed on determination and optimization of the following operating conditions: operating temperature of shaft furnace or electric arc furnace (temperature of reduction smelting); required carbon amount; required amount and composition of fluxes.

According to the chemical analysis of red mud and conducted thermodynamic analysis using HCS Chemistry, presented on the Ellingham diagram (Fig. 2), carbon oxidation to CO can lead to reduction of H_2O , Fe_2O_3 , Na_2O and V_2O_5 at the temperatures above 900 °C and to reduction of SiO_2 and TiO_2 at the temperatures above 1700 °C, while Al_2O_3 и CaO can't be reduced at the temperatures lower than 2000 °C. The main chemical reactions, occurring during pyrometallurgical reduction of red mud by carbon are presented below:



As soon as the melting temperature of clean Fe makes 1537 °C and the cast iron melting temperature is within the range 1150–1200 °C (depending on cast iron composition), the furnace operating temperature should be located within

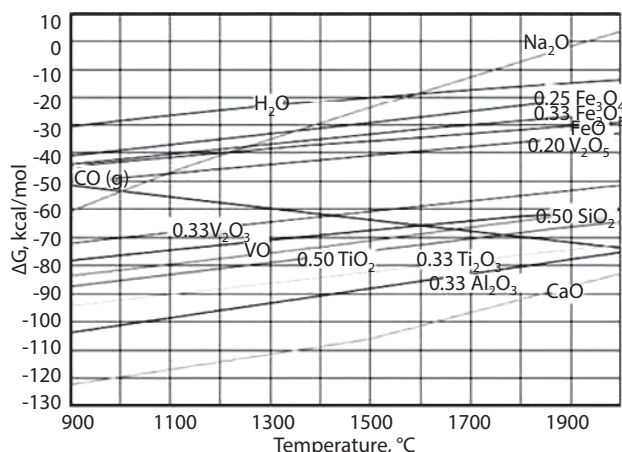


Fig. 2. Ellingham diagram calculated using HSC Chemistry 6 software

the range $1300\text{ }^{\circ}\text{C} < T < 1650\text{ }^{\circ}\text{C}$, to provide efficient decrease of Fe content in red mud and to avoid at the same time lowering of silicon and titanium content [18]. Based on analysis of this research, slag which was obtained after reduction smelting, has liquidus temperature above $1450\text{ }^{\circ}\text{C}$; thereby the furnace operating temperature was set for this investigation at the level $1450\text{--}1500\text{ }^{\circ}\text{C}$.

To take into account the accompanying reactions, which consume carbon, reduction smelting was examined in the work [16] at 4 different initial carbon concentrations: stoichiometric carbon (e.g. carbon is required in stoichiometric mode for complete Fe and H_2 reduction in 1 kg of red mud); carbon with excess in relation to stoichiometric carbon by 5 %, 7.5 % and 10 %. It was concluded from this theoretical investigation that carbon excess at least 7.5 % is required for sufficient covering of all accompanying and secondary reactions occurring in the system and thereby for achievement of high degree of Fe extraction from red mud. CO and H_2 dominate in the gas phase, while absence of SO_2 in gas phase manifests that sulfur remains completely in the slag and metal phases. Additionally, small amount of Na vapours is predicted, despite 58 % of Na remains in the slag phase as oxide. However, slag obtained in such conditions is strongly alkali slag (basicity relation >1.8), what is considered as a essential deficiency for the process realization due to potential destruction of furnace lining. This slag is also characterized by rather high liquidus temperature ($> 1450\text{ }^{\circ}\text{C}$) and low viscosity, what makes complications for its use in cement production.

It is also important, that is suggestion about complete transition of radioactive elements (containing in red mud) in the slag phase is true, then obtained slag will have high radioactive composition (activity concentration index $I = 9$), what makes it useless for application as a typical building material (e.g. mineral wool). This was the reason, why additional technological flows (SiO_2 и CaO) were added in the research [16] in order to dilute concentration of radioactive elements. Based on calculations, at least 220 kg of fluxes per ton of processed red mud is required to achieve $I < 6$.

In this case the liquid slag phase, which was calculated using Factsage program for different total amount of additives and different mass relationship CaO/SiO_2 (basicity), will form at the temperature below $1400\text{ }^{\circ}\text{C}$ and, respectively, is suitable both for steel making and steel consequent application.

Taking into account economical substantiation of the process, which can be complicated by adding of large amount of fluxes or production of high-aggressive slag (acid or alkali) that can damage furnace lining and equipment, it is suggested to examine in this work also use of totally 350 kg of fluxes per 1 ton of red mud, with total mass relationship CaO/SiO_2 within the range 0.8-1.0.

According to the chemical analysis of red mud and the Ellingham diagram, carbon oxidation in CO can lead to reduction of H_2O , Fe_2O_3 , Na_2O and V_2O_5 at the temperatures above $900\text{ }^{\circ}\text{C}$, as well as SiO_2 and TiO_2 at the temperatures above $1700\text{ }^{\circ}\text{C}$, while Al_2O_3 and CaO can't be reduced at the temperatures lower than $2000\text{ }^{\circ}\text{C}$. As soon as the smelting

temperature of clean Fe makes $1537\text{ }^{\circ}\text{C}$, the steel smelting temperature is within the range $1150\text{--}1200\text{ }^{\circ}\text{C}$ (depending on its composition), and the furnace operating temperature should be from 1300 to $1650\text{ }^{\circ}\text{C}$ to provide efficient Fe content in red mud, avoiding lowering of silicon and titanium content. Based on the work [17], slag obtained after reduction smelting has the liquidus temperature above $1450\text{ }^{\circ}\text{C}$, thereby and the furnace operating temperature was established for this research at the level $1450\text{--}1500\text{ }^{\circ}\text{C}$.

Residual Fe content $< 0.2\text{ }%$ (mass.) was obtained for all slags. Degree of Fe extraction in cast iron was evaluated according to the Fe content in slag and cast iron (obtained according to the data of X-ray structural analysis); in this case we accept that Fe-containing phases are not presented in sublimes and in dust. Based on this calculation, maximal Fe extraction in cast iron was obtained at the temperature $1450\text{ }^{\circ}\text{C}$ and makes 88.5 % and 91.5 % in average (in the case of fluxes use without and with soda respectively). Composition of cast iron obtained via reduction smelting is presented in the **Table 3** and the results of cast iron structure examination — on the **Fig. 3**.

According to the results of X-ray structure analysis, cast iron contains 93.8 % (mass.) Fe and 4.99 % (mass.) C. Cast iron structure is considered as hyper-eutectic white cast iron (ledeburite + graphite). Non-metallic inclusions were not found out in structure of samples.

All obtained slags are viscous and extremely hard. Soda introduction in charge allows to lower the liquidus temperature and slag viscosity, what improves metal coagulation and metal phase separation with slag extraction, as well as slag tapping and consequent comminution (slag with soda addition is less hard).

Most slags have small sodium content based on the results of X-ray structural analysis (**Table 4**, sodium extraction in slag makes 13-19 %), what testifies on dominating transition of sodium-containing phases in sublimes during reduction smelting. It means that soda addition in charge leads to increase of consumption of reacting agents, wear of equipment owing to its corrosion and large amount of alkali emissions.

The results of differential thermal analysis (DTA) of red mud, obtained after reduction smelting, display that no phase transformations are observed in the sample below the temperature $1000\text{ }^{\circ}\text{C}$ and mass losses are small. Thermogram analysis shows only one peak below the temperature $100\text{ }^{\circ}\text{C}$ which can correspond to loss of hygroscopic moisture. It should be noted that compositions of slags, obtained after reduction smelting without and with charge addition, are similar.

Based on analysis of technical literature and the researches conducted by the authors of this article, the large-scale technological route for processing of red mud was developed; it includes pyrometallurgical method of Fe extraction in cast iron via reduction smelting with carbon, with evaluation of the main production material flows (**Fig. 4**). If consumption of processing raw materials makes 2 mln. tpy, then cast iron in amount of 503.376 thousand tpy will be produced, according obtained material balance within the technological line of preliminarily preparation and reduction smelting of

Table 3. Chemical composition of obtained cast iron

Component	Fe	C	Si	P	S	Mn
Content, % (mass.)	93,8	4,99	0,01	0,33	0,03	0,37

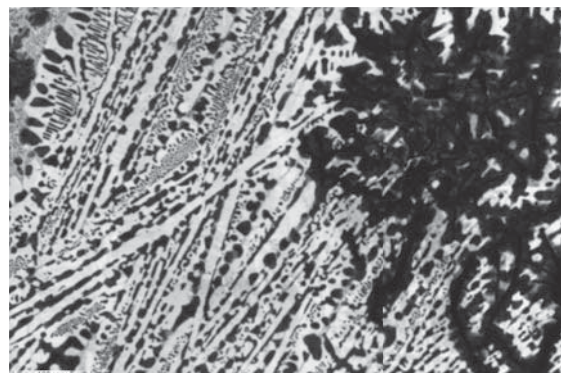
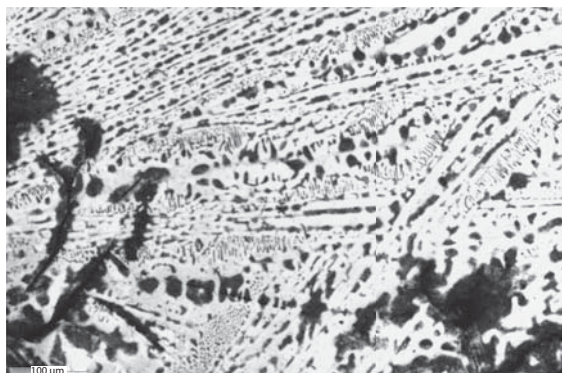


Fig. 3. Cast iron structure, obtained via reduction smelting, ×200

Table 4. Slag chemical composition

Processing method of red mud	Content of the elements, % (mass.)							
	Fe	Al	Ti	C	S	Na	Ca	Si
Initial red mud	28.15	8.35	2.36	1.20	0.71	2.12	6.29	7.12
Reduction smelting without soda	0.22	13.71	2.94	1.97	0.51	0.26	28.70	10.67
Reduction smelting with soda	0.16	13.54	2.95	1.41	0.51	1.82	27.64	11.23

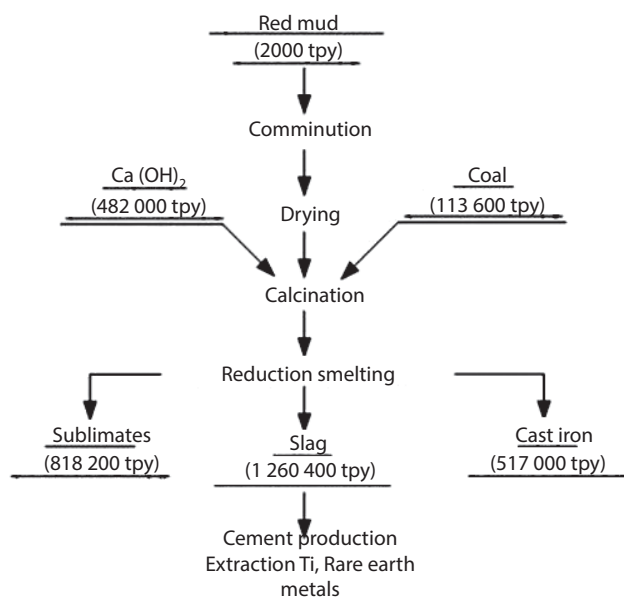


Fig. 4. Material flows of cast iron production route from red mud

red mud. As well slag will be also produced, which concentrates oxides of aluminium, titanium and rare earth metals in amount of 1,200.4 thousand tpy. In accordance with the preliminary technical and economical analysis, production profitability for this production volume makes 36.7 % and payback period – 2.7 years.

Reduction smelting with carbon allows to extract up to 99 % (mass.) of Fe from red mud in cast iron. Slag with residual Fe content (less than 1 % (mass.)) can be forwarded to extraction of alumina and titanium (average alumina content in slag makes 26 % (mass.), silica and Fe₂O₃ content 22 % (mass.) and only 0.3 % (mass.) respectively).

Conclusions


1. Based on the results of conducted researches and analysis of technical literature, it is suggested to extract Fe in cast iron via reduction smelting with carbon. Maximal Fe extraction in cast iron was obtained at the temperature 1450 °C and made in average 88.5 % and 91.5 % for the cases of using fluxes without and with soda addition respectively. According to residual Fe content in slag (based in the data of X-ray structural analysis), Fe extraction in cast iron exceeds 99 %.

2. Temperature rise up to 1470–1480 °C and increase of holding duration promote essential elevation of losses of valuable elements and lowering of metal extraction degree. On the contrary, decrease of the temperature down to 1420 °C also leads to lowering of metal extraction degree, perhaps due to increase of slag viscosity and density, what complicates the processes of metal separation and coagulation and also slag tapping.

3. Almost 100 % (about 99.6-99.9 %) of aluminium passes into slag; also silicon (about 85-90 %) and calcium concentrate in slag. Titanium extraction in slag makes in average about 80 %. Alkali metals practically completely pass

in sublimates. Sulfur passes in slag partly (about 50 %), its rest part passes in sublimates.

4. All obtained slags are viscous and extremely hard. Soda introduction in charge allows to decrease liquidus temperature and slag viscosity, what improves metal coagulation and metal phase separation from slag, as well as slag tapping and consequent comminution (slag with soda addition is not so hard). However, small amount of sodium (sodium extraction in slag makes 13-19 %) was revealed in most slags based on the results of X-ray structural analysis; it testifies on dominating transition of sodium-containing phases in sublimates during reduction smelting. It means that sodium addition in charge leads to increase of consumption of reacting agents, wear of equipment owing to its corrosion and large amount of alkali emissions.

5. Technological route for processing of red mud with Fe extraction in cast iron is suggested in accordance to the above-mentioned results; economical expediency of this method is shown. 

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