

On the technology of processing of manganese wastes from ore concentration: metallization of granulated manganese pellets via low-temperature roasting

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The proposed technology for processing of manganese-containing wastes from ore concentration represents a new approach to obtaining low-carbon ferromanganese from wastes. Based on the detailed comprehensive studies of two interrelated stages of the common technology (preparation of granulated pellets from a charge of preset composition and their consequent processing via low-temperature roasting), possibility of obtaining metallized granulated pellets is shown in this research. The main regularities of iron and manganese reduction during roasting within the temperature range 773–1800 K are established. Based on the comprehensive analytical studies of the material composition and forms of manganese, which were carried out using X-ray phase analysis, scanning electron microscopy and gas chromatography, step-by-step quality control of the products obtained during implementation of the technological cycle of low-temperature roasting is ensured. It was found out that beginning of abundant CO release in the gas phase under conditions of low-temperature roasting is achieved at a temperature ~1373 K. Temperature rise increases the CO content in the gas phase. Maximal CO content in the gas phase is 94 % CO, it was reached at the temperature of ~1473 K. It is displayed that increase of CO content in the gas phase is mainly determined by the course of reactions of wustite and magnetite reduction by carbon. Mn_3O_4 reduction reaction is hindered due to the high chemical strength of the oxide. It is indicated that higher roasting temperatures (~1773 K) are required to intensify reduction of strong manganese oxides (Mn_3O_4 , MnO) by solid carbon. As a result of low-temperature roasting of granulated manganese pellets, metallized pellets with content of 23.67 % Fe_{gen} ; 23.2 % Fe_{Me} ; 0.7 % Mn_{Me} ; 28.47 % MnO ; 5.0 % C etc. were obtained.

Key words: manganese wastes, mono-charge, granulation, low-temperature roasting, thermodynamic analysis, Gibbs energy, carbon, temperature, metallized manganese pellets.

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Introduction

Increase of global steel production rises demand on rich ores and concentrates. Based on the data of the World Steel Association (Worldsteel), 148.8 mln. t of steel was smelted in 71 countries, which supply Worldsteel with their statistical information; in exceeds indicator of the same month of 2023 by 3.7 %. Daily production of smelted steel was 5.131 mln. t [1]. Depletion and degradation of rich ores creates global tendency for increase of ferroalloys production cost.

The world practice of ferroalloys production is based on reduction melting of charge, which consists of lump ores, coke and flux, with large consumption of reducing agent and electric energy for charge heating and melting [2]. Fine wastes are forming during classification of initial agglomerated raw material, they can't be recycled for secondary processing via conventional technologies. Preparation for agglomeration of fine wastes is accompanied by definite expenses. Their return to the conventional technological cycle is possible in such extent, in which they can be sub-

jected to agglomeration with maximally low expenses for this preparation.

Sintering is considered as the most widely distributed method for agglomeration of fine ore materials and concentrates; it is realized at the temperature 1723 K. Fine particles can't even be close to softening state, because the process is ensured at high temperatures 1873–1973 K [3, 4]. Usual sintering machines can't operate at such temperatures. At present time, sintering is not considered as an effective process, both from technological and environmental sides.

Agglomeration with manufacture of pellets is the second widely used method for preparation of fine-dispersed material before processing. This process requires essential expenses for comminution of fine wastes to dispersed state, for consumption of binding materials and for use of additional equipment [5].

Iron making and steelmaking as well as ferroalloys production are very responsible industries in greenhouse gases at present time. Importance of solving this problem is more strong based on the experts' forecasts that expected perma-

ment emission of anthropogenic greenhouse gases can lead to harsh change of climatic conditions [6]. It is evident that putting into practice of breakthrough technologies and use of alternative fuel (depleted by CO_2) should be applied for cutting the emissions. Use of biomass, biocarbon of hydrogen as reducing agents can be possible variants for solving this problem. Carbon on biological base can be considered as CO_2 -neutral, if the same amount of biomass is consuming and reclaimed [7]. Consequently, replacement of carbon fossils e.g. by charcoal can lead to substantial decrease of specific CO_2 emissions in the atmosphere [8].

Searching the methods for processing of fine-dispersed wastes from concentration of chromium and manganese ores is considered as the separate problem. These wastes are accumulated in the volumes exceeding the critical level of their influence on subsoil waters, aqueous flora and environment [9–11].

There are several researches presented in the scientific literature, which are devoted to effective solving the problem of processing of fine-dispersed chromium- and manganese-containing wastes [12–14]. However, putting into practice of such wastes is restricted and is not widely used due to high capital expenses and strict ecological requirements, which are linked with large emissions of greenhouse gases.

The results of the research [15] present serious theoretical and practical interest; it describes the new approaches to technologies of steelmaking and ferroalloys production, which are based on the common calculation technique for initial charge with varying composition. The authors proposed technical solution of extraordinary interest from the point of view of involving fine-dispersed chromium- and manganese-containing wastes after mineral processing of corresponding ores and other multi-component raw materials (different in their kinds and compositions) in production, with high extraction of valuable metals from these materials. Use of charcoal as a reducing agent instead of carbon in the developed technology makes it especially attractive.

The concept of the developed technology includes step-by-step solving of the following tasks:

- (1) preparing of the complex mixture (mono-charge) with varying composition on the base of manganese-containing wastes and manufacture of pellets from this mixture;
- (2) production of metallized manganese pellets with preset composition via low-temperature roasting of preliminarily prepared pellets;
- (3) reduction of metallized pellets in and electric furnace with obtaining ferromanganese, excluding conventional processes of metal carbonization and ironmaking.

The problems of preparing of mono-charge with varying composition (1) on the base of manganese-containing wastes and granulated pellets are described in the work [16] in details.

The aim of this research is examination of the forming mechanism for metallized granulated manganese pellets during low-temperature roasting.

The process of preparing of metallized granulated manganese pellets with preset composition is an intermediate stage (2) for conduction of consequent reduction melting of these pellets (3), within the framework of the concept for building a common technology of processing of manganese-containing wastes after ore concentration with obtaining of commercial ferromanganese.

Materials and methods of the research

Materials. Granulated pellets, which were prepared from complex mixture (mono-charge) with preset composition, were used for carrying out the researches [16]. Composition of pellets was identical to the average composition of a complex mixture. Accumulated wastes from concentration of manganese ores from Dzhezdinsky mining and concentrating plant, rolling mill scale from steelmaking facilities of “Arcelor Mittal” and charcoal obtained via pyrolysis of biological wastes (starting from agrestal grass to agricultural wastes, such as straw, cotton stems etc.) were taken as the base for mono-charge obtaining.

Chemical composition of initial charge components are shown in the **Table 1**. Nass relationship between manganese wastes and rolling mill scale makes 0.9:0.1, what ensures obtaining of metallized pellets during reduction roasting [17]. The average chemical composition of the charge is displayed in the **Table 2**.

Research methods. Theoretical description of metallurgical processes, occurring in the conditions of low-temperature charge roasting (within the temperature range 773–1473 K) with obtaining Fe-metallized granulated pellets is the main methodological principle of this technology.

Investigations of solid phase reduction of Fe and Mn oxides by carbon were conducted on the base of thermodynamic analysis of their interaction reactions within the temperature range 773–1873 K.

Calculations included determination of Gibbs free energy loss from reactions depending on the temperature. Thermodynamic calculations were carried out using software HSC Chemistry 8.1.5 of the company Outotec Technologies, taking into account the results of substantial analysis

Table 1. Chemical composition of initial charge components

Material	Content, % (mass.)										
	Fe	FeO	Mn	SiO_2	Al_2O_3	CaO	MgO	S	P	C	Other
Mn withdrawal	11.21	–	28.35	32.25	4.20	1.78	0.55	0.8	0.21	–	20.65
Rolling mill scale	66.67	32.21	0.44	–	–	–	–	0.01	0.50	–	0.17
Charcoal	–	–	–	1.02	0.26	–	–	–	0.37	98.0	–

Table 2. The average chemical composition of the charge [16]

Elements	Fe	Mn	SiO_2	Al_2O_3	CaO	MgO	S	P	C	Other
Content, % (mass.)	11.94	18.67	20.46	4.21	17.10	0.73	0.58	0.02	11.40	14.89

and forms of Fe and Mn presence in charge components and roasting products.

Elementary compositions of initial charge components and obtained products were determined using mass spectrometer Agilent 7700 Series ICP-MS (USA) with inductive connected plasma, optical emission spectrometer Optima 2000 DV of the company Perkin Elmer SCIEX with inductive connected plasma as well as gravimetric and chemical analytical sensors.

X-ray phase analysis of samples was conducted using the sensors D8 Advance (Bruker AXS GmbH), α -Cu, with voltage 40/40 in an X-ray tube. Processing of the obtained diffraction patterns and calculation of interplane distances were carried out using EVA software. Decoding of samples and searching of phases were implemented using the program Search/match with use of ASTM card database. The error of semi-quantitative analysis makes $\pm 5\%$.

Crystal optic analyses of samples were carried out using scanning electron microscope and such microscopes as Olympus BX-51 and Leica DM2500.

The results of substantial composition of initial materials, which make the base of granulated mono-charge and forms of manganese presence in wastes after concentration of manganese ores, are described in details in the researches [16, 17].

Experimental part

The scheme of laboratorial unit and the technique of experiments

Low-temperature roasting of granulated manganese pellets was carried out in an electric furnace at the temperature 1473 K.

In order to accumulate initial material for preparation of metallized pellets, 11 experiences were conducted at the same conditions.

Charge granulation was executed in a disk granulator, where molasses solution was used as a binding component; this solution was introduced in the amount of 3 % of molasses for a mixture of wet material. The obtained material with fraction 8.0–20.0 mm was subjected after granulation to drying at the temperature 673 K in a drying furnace. Solid carbon in composition of a complex mixture was not oxidized practically in the air atmosphere at the preset temperature, and it does not react with Fe and Mn oxides.

The granules with diameter 8.0–20.0 mm were obtained after drying and they were used consequently in low-temperature reducing roasting.

The experiments were carried out with initial charge of pellets in the amount 200 g, with roasting duration ~ 90 min. Experiment finishing was determined by termination of gases (CO , CO_2) output. The scheme of the laboratorial unit is shown in the Fig. 1.

The technique of conducted experiments concluded in the following operations. The crucible with initial charge of pellets 3 was charged in the electric furnace 1 SUOL-044 12-M2 (preliminarily heated up to 773 K). Then the furnace was heated to 1453 K with heating rate $10\text{--}12^\circ\text{C}/\text{min}$. Temperature control and varying were implemented using a thermocouple PP-1 6 and a secondary sensor KSP-4 13.

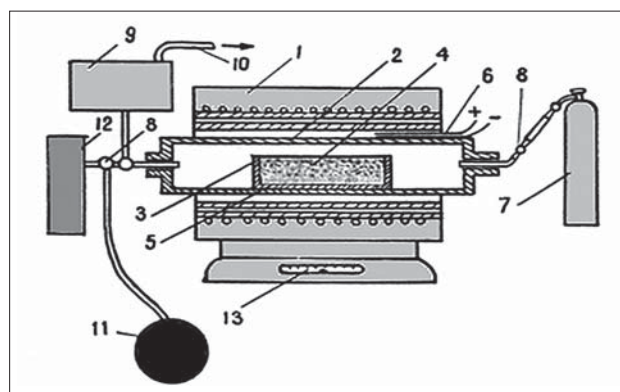


Fig. 1. The scheme of the laboratorial unit for conduction of low-temperature roasting of granulated manganese pellets: 1 – electric resistance furnace; 2 – quartzite tube; 3 – crucible with charge; 4 – granulated pellets; 5 – crucible; 6 – thermocouple PP-1; 7 – reservoir with argon; 8 – three-way valve; 9 – gas flowmeter SGK-G4; 10 – gas exhaust; 11 – gas chamber; 12 – gas chromatograph; 13 – secondary sensor KSP-4.

Gas withdrawal from the reaction area of the furnace was realized by argon flow from reservoir 7. Argon consumption was 100 ml/min. Total gas volume, which was extracted from the furnace, was fixed using a flowmeter SGK-G4 (manufactured by the Instrument-making corporation “Signal”, Russia) 9 and was then collected in the gas chamber 11. Gas composition was monitored for CO and CO_2 content via gas chromatograph “Kristall 2000M” 12. When analysis is finished, gas is withdrawn in the atmosphere using exhaustor 10.

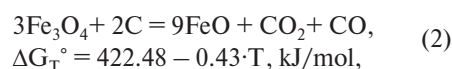
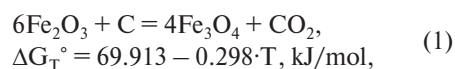
When the preset temperature is achieved, the charge in the furnace was held until stopping extraction of gas products (~ 90 min). Gas extraction from the furnace was completely terminated during the preset time period, what meant finishing of reactions of solid phase reduction of metals from their oxides by carbon and complete charge metallization.

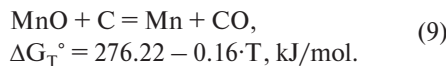
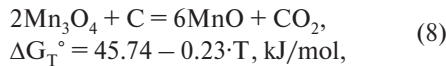
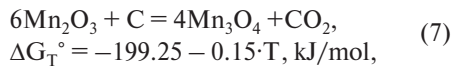
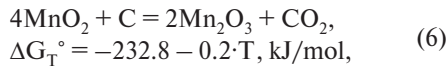
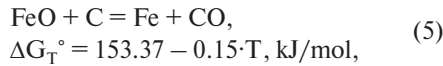
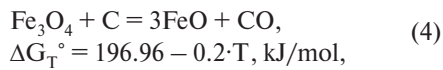
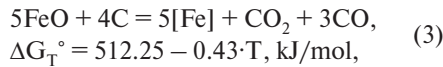
After finishing of each experiment, pellets were unloaded from the furnace, weighed and sent for analysis. All granulated pellets, which were collected after conduction of 11 experiments (~ 2 kg) were used for carrying out their reduction melting in order to obtain ferromanganese.

Results and discussion

Thermodynamic analysis of interaction reactions between higher and lower Fe and Mn oxides with solid carbon is the base of the research of pellets components behaviour in the conditions of low-temperature roasting. The selected temperature range $700\text{--}1800$ K covers the area of low-temperature roasting, applying to metallization of granulated manganese pellets.

The reduction mechanism for iron and manganese oxides during low-temperature roasting can be presented as the system of the following reactions:





The relationship between free Gibbs energy (ΔG_T°) and the temperature of the reactions (1)–(9) is displayed in the Fig. 2, 3.

The results of thermodynamic calculations show that phase transformation of hematite (Fe_2O_3), which corresponds to its reduction by carbon via the reaction (1), starts

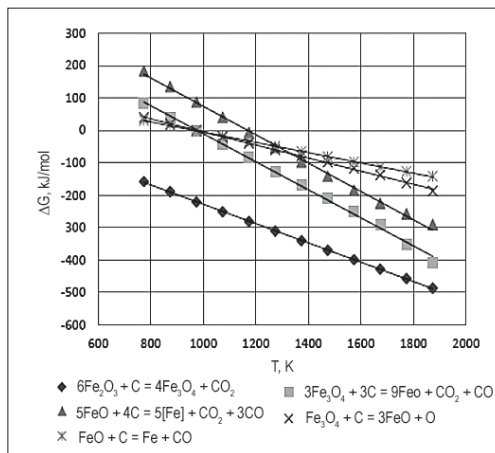


Fig. 2. Variation of free Gibbs energy for the reactions (1)–(5) depending on the temperature

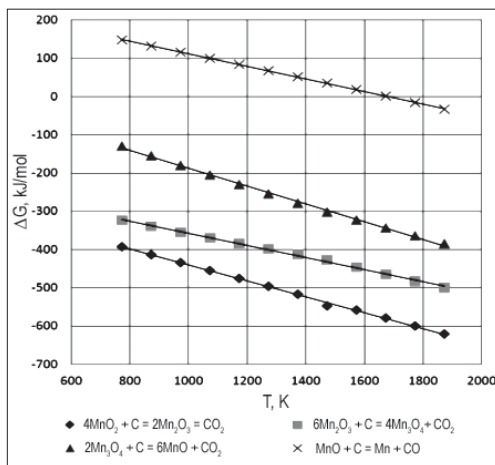


Fig. 3. Variation of free Gibbs energy for the reactions (6)–(9) depending on the temperature

just at the temperature ~ 773 K, $\Delta G_{773\text{K}} = -158$ kJ/mol. Further temperature rise strengthens the reaction (1) and achieves $\Delta G_{773\text{K}} = -370.5$ kJ/mol for the roasting temperature 1473 K (see Fig. 2).

Reaction (1) finalizes in forming of magnetite, which then interacts with carbon via the reactions (2) and (4) and transits in a stable lower iron oxide – wustite FeO. These reactions start at the temperature 973 K and their rate increases with temperature rise (see Fig. 2). High values of free Gibbs energy for these reactions testify on this observance: their values make $\Delta G_{1473\text{K}} = -207.4$ kJ/mol and $\Delta G_{1473\text{K}} = -98$ kJ/mol respectively for the reactions (2) and (4) at the temperature 1473 K.

Possibility of reduction of strong wustite to metallic iron by carbon via the reactions (3) and (5) is established. It is noted that wustite reduction with stoichiometric carbon consumption starts a little earlier than at the temperature 973 K ($\Delta G_{973\text{K}} = -0.63$ kJ/mol). When carbon consumption is excessive, passing of the reaction (5) is impossible at the preset temperature, owing to the positive value of free Gibbs energy, $\Delta G_{973\text{K}} = 87.71$ kJ/mol. Passing of the reaction (5) starts at the temperature 1073 K; this conclusion is very important for the practice. When ferromanganese is manufactured from preliminarily prepared charge, with its solid phase reduction, presence of excessive carbon consumption is not required.

The mechanism of phase transformation for higher manganese oxides with obtaining manganese metallic state during roasting is passing in another way.

We can see from the Fig. 3 that higher manganese oxides start to be reduced by carbon with obtaining its solid oxide MnO, according to the reactions (6), (7) and (8) at the temperature 773 K. The values of free Gibbs energy for these reactions at this temperature are equal to: $\Delta G_{773\text{K}} = -392.8$ kJ/mol; $\Delta G_{773\text{K}} = -324$ kJ/mol and $\Delta G_{773\text{K}} = -129.6$ kJ/mol respectively, and they exceed seriously the values of free Gibbs energy for the reaction (1) – hematite reduction by carbon. However, further MnO reduction to metal is practically impossible, what is confirmed by the positive values of free Gibbs energy for the reaction (9) up to the temperature 1773 K. Reduction of lower strong manganese oxide is possible only at the high temperatures above 1773 K (see Fig. 2).

The mechanism of behaviour of Fe and Mn oxides in the conditions of low-temperature reduction charge roasting can be formulated after analysis of the obtained results. This charge was prepared on the base of manganese wastes and solid carbon. Fe is completely reduced from its oxides to metallic state during roasting. Higher manganese oxides are completely reduced to their strong oxide MnO and its further reduction to metallic manganese in the conditions of low-temperature roasting is practically impossible. As a result of roasting, metallized material, which is ready for further reduction melting at high temperatures, is forming.

The established regularities are completely confirmed by the results of laboratorial experiments. Chemical composition of the obtained metallized manganese pellets after roasting (see Table 3) displays that complete Fe reduction to metallic state is achieved during roasting. Mn reduc-

Table 3. Chemical composition of metallized manganese pellets

Components	Fe _{tot}	Fe _{Me}	Mn _{Me}	MnO	SiO ₂	Al ₂ O ₃	MgO	C	Other
Content, %	23.67	23.2	0.70	28.47	28.4	3.7	0.48	5.0	13.62

tion does not take place in this case; manganese is presented in finished metallized pellets mainly by its lower oxide MnO. Such impurities as sulfur and phosphorus are completely removed with gases during roasting.

Increased carbon content (5.0 %) in obtained pellets is not critical, because it will be used completely for MnO reduction during consequent reduction melting.

Total gas output and gas extraction rate are shown in the Fig. 4 in the experimental conditions, depending on temperature rise.

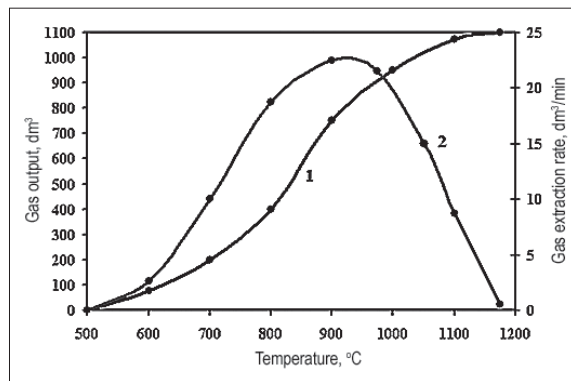


Fig. 4. Varying of total gas output (1) and gas extraction rate (2) depending on temperature rise

Relationship between the gas phase composition and the temperature is presented in the Table 4.

It can be seen from the Fig. 4 that extraction of the gases (CO+CO₂) during experiments was observed at the temperature 773 K, what testified about start of metal reduction reaction by carbon. The fast growth of total output of gases is seen at the temperature above 873 K (Fig. 4, curve 1); it increases until the end of this experiment, with varying CO and CO₂ composition. Typical strengthening of total gas output, which is accompanied by increase of CO₂ concentration in the gas phase, is presented within the temperature range 873–1173 K (what can be surely explained). The essence of the established regularities can be interpreted in the following way.

Firstly, high iron oxide (hematite), which is characterized by lower strength, begins to interact with solid carbon via the reaction (1). Taking into account the fact that elasticity of

dissociation of lower iron oxides Fe₃O₄ and FeO within the temperature range 873–1173 K is rather high ($\text{Lg}P_{\text{O}_2} = -20.8$ for Fe₃O₄; $\text{Lg}P_{\text{O}_2} = -22.3$ for FeO (P_{O_2} , atm.)), we can wait for iron reduction from the a.m. oxides by solid carbon with CO₂ and CO forming via the reactions (2) and (3).

The basic growth of CO₂ content in a gas phase provided passing the reactions (1) and (2). Passing of the reaction (3) is insignificant, due to FeO binding in silicate. Based on this, it is easy to observe the order of metals reduction within the temperature range 873–1173 K: at first maximal reduction of higher iron oxides takes place, while manganese oxides only begin to be reduced by solid carbon.

Analysis of gas composition shows achieving of high (up to 90 %) CO₂ concentration in gas at the temperature 1173 K (see Table 4), what is connected by passing the reaction (1), as it was mentioned before. Fast growth of gas extraction rate within the temperature range 873–1173 K testifies on intensive passing of the reaction (1). Maximal value of CO₂ content in a gas phase (24 dm³/min) was achieved at the temperature 1173 K (see Fig. 4, curve 2).

At the high roasting temperatures (above 900 °C), the processes of reduction of strong iron oxides (Fe₃O₄, FeO) by carbon via the reactions (4) and (5) will be overlapped consequently in addition to the reactions (1)–(3).

Conduction of the reactions (2)–(5) increases CO concentration in a gas phase due to lowering of CO₂ concentration. This regulation is completely confirmed by the results of experiments, which are presented in the Table 4 and in the Fig. 4.

Start of abundant CO extraction in a gas phase was recorded experimentally at the temperature ~1373 K (see Table 4). The following temperature rise leads to increase of CO content in a gas phase, which achieved its maximum equal to 94 % CO at the temperature ~1473 K.

Increase of CO content in a gas phase is mainly determined by passing of the reactions (4) and (5). Passing of the reaction (5) is not so important, due to FeO binding in silicate. Manganese oxide reduction is more preferable via the reaction (7). Passing of the reaction (8) is hindered due to high chemical strength of Mn₃O₄ oxide, and it provides only a slight input at the temperature ~1473 K. To intensify the reduction processes for strong manganese oxides (Mn₃O₄, MnO) by solid carbon, higher temperatures are required.

Table 4. Varying of the gas phase composition depending on temperature rise

Time, min	Temperature, K	Gas phase composition, %		Total degree of charge reduction
		CO ₂	CO	
11	923	55.0	45.0	0.053
23	1003	66.0	34.0	0.173
36	1123	73.0	27.0	0.344
50	1173	88.0	12.0	0.628
65	1353	18.0	82.0	0.746
80	1373	12.0	88.0	0.821
90	1453	6.0	94.0	0.834

It can be seen from the Fig. 4 (curve 2), the gas output rate above the temperature 1173 K is gradually decreasing to zero, what testifies on maximal iron oxide reduction by solid carbon. Reduction of manganese oxide in this case is minimal, what is confirmed by the chemical composition data for obtained metallized pellets.

Thus, the results of laboratorial experiments completely confirm the conclusions of thermodynamic calculation and display principal possibility of metallization of manganese granulated pellets in the conditions of low-temperature roasting.

Chemical composition of obtained metallized pellets (see Table 3) shows practically complete (up to 98 %) iron reduction by solid carbon.

The technology for preparation of mono-charge on the base of accumulated wastes from concentration of manganese ores is developed for supplying Aktyubinsk and Aksus ferroalloy plants. Until present time, accumulated manganese-containing wastes were not used effectively. Now this disadvantage is corrected by preparing metallized pellets on the base of these wastes, and such pellets can be subjected to melting with manufacture of commercial ferromanganese without any serious expenses.

The results of these researches will be used for preparation and efficient processing of manganese fine wastes at Zhesdinsky mining and concentrating plant in Kazakhstan for production of high-quality ferromanganese, which is required at domestic and international markets. The feature of this technology concludes in elimination of coke consumption, lowering of electric power consumption, decrease of gas emission in the atmosphere and (what is rather important) in rise of economic and ecological efficiency.

Conclusion

Unlike the conventional technology, the suggested development is based on the original technique of charge preparation from accumulated fine-dispersed manganese wastes. These fine-dispersed wastes were accumulated during concentration of manganese ores and made the base of mono-charge in the conducted investigations.

The developed technology of ferromanganese production includes preparation of mono-charge with introduction of dosed coal amount in its composition for reduction of extracted metals (Fe and Mn), depending on concentration of their oxides in initial raw materials, as well as introduction of rolling scale, which allows increasing of Fe content in charge. After mixing and comminution, granulated manganese pellets are prepared, which are subjected to low-temperature roasting for obtaining metallized pellets. Obtained pellets are then subjected to consequent reduction melting until obtaining high-quality ferromanganese.

The positive results of thermodynamic calculations, which were confirmed by the data of laboratorial investigations, display possibility of effective processing of manganese-containing wastes and will be applied in the future during conduction of laboratorial researches for processing of multi-component manganese-containing raw materials and wastes of metallurgical works and concentrating plants (which are complicated in their compositions and kinds). 

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