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ХИМИЧЕСКАЯ ТЕХНОЛОГИЯ
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Research of brown iron lisakovsky raw materials solid phase metallization process

The research results of the process of lisakovsky gravitational magnetic concentrate (LGMC) solid-phase metallization using the highly reactive solid carbon reducing agent «direksil» are given in the article. There were set rational parameters of solid-phase reduction process of LGMC in the mine furnace providing the extent of its metallization of 20.4–88.6 %. It was determined the temperature range of solid-phase direct reduction of iron of 1150–1200 ºC providing the maximum rate and extent of iron oxides metallization, at the same time passing over emergence of the liquid phase — fusion. Use of «direksil» as a reducing agent allows raising metallization extent of LGMC in more than 3 times in comparison with furnace coke. In the article there are given diffraction patterns (X-ray patterns) of the metallized samples of LGMC characterizing phase structure and crystal structure of the obtained restoration products depending on the type of reducing agent. The physical condition of the metallization products obtained in the mode of solid-phase restoration allows using all known methods of separation of the metallized concentrate from the dead rock. The most preferable option of such separation includes methods of magnetic separation in view of contrast formed after the system metallization. Separation of magnetic and non-magnetic products after metallization is an obligatory element of the technological scheme of LGMC metallization since the total content of iron in initial concentrate does not exceed 49 %. At that depending on metallization extent the magnetic fraction can be used as a furnace charge component when smelting the conversion iron in the blast furnace (metallization extent is less than 40 %) or steelmaking in the electric furnace (metallization extent is more than 80 %).

Keywords: brown iron, LGMC, solid-phase metallization, metallization extent, carbon reducing agent, direksil, phase structure, metallized concentrate.

Many research works and publications are devoted to studies of the iron restoration process from the lisakovsky gravitational and magnetic concentrate (LGMC) during a number of decades [1–6]. In particular the scientists of Zh. Abishev Chemical and Metallurgical Institute are engaged in solution of the problem of benefication and refinement (dephosphorization) of LGMC [3–6] for many years. In the works [2–6] there are concerned the results of reducing roasting of LGMC with use of gaseous, solid and liquid reducers. Authors [2] established that restoration rate of concentrate's layer was limited by external diffusion at designed temperature and gas composition. And, as the studies have shown, the external diffusive resistance for a concentrate is much less than internal diffusive resistance, for example, for the iron nugget concentrate, and is regulated by change of gas consumption, due to which the metallization rate increases significantly. This advantage of the concentrate over the layer of lumpy materials can be used for solution of problems of direct metallization of iron ore concentrates, in particular, in vertical porous tanks. But the main obstacle in the way for realization of such process is susceptibility of usual iron ore concentrates to adhesion and swelling during restoration. However, according to authors [2], as for metallization of LGMC, the process proceeds without adhesion of particles even at 1000 ºC. Lisakovsky concentrates in the whole interval of reduction degree from 0 to 1 do not conglutinate both at low and high (1000 ºC) temperatures, they do not inflate and, on the contrary, considerably shrink. These features of them are favorable to solution of tasks on solid-phase metallization, with expansion of temperature limits of reduction in order to achieve the high rate of interaction. To overcome the difficulties connected with low burdening materials' bed porosity it is necessary to create such conditions for metallization of ore raw materials at which the rate of the process would not be limited by diffusive resistance. It is possible to solve this important problem when using reducer with the high reactive capacity and applying the special technology on preparation of burden constituents.

The problem of the real researches consisted in determination of temperature limits of solid-phase direct reduction of iron with a view to provide the maximum rate and extent of metallization of iron oxides, at the same time passing over emergence of the liquid phase — fusion. For solution of that we have conducted a series of experiments on studying the influence of temperature condition on metallization of the lisakovsky gravitational and magnetic concentrate in the heat-treatment furnace (mine furnace). Test activity on direct restoration was carried out at 1000–1250 °C temperatures. The furnace coke (Arselor Mittal Temirtau) and new highly reactive reducer «direksil» were used as reducing agents. Carbonaceous reducing agent «direksil» was obtained from the enriched nonlinkering coal by the method of high-speed thermal-oxidative carbonization. Development and exploitation of the production technology of direksil were carried out under the direction of professor, laureate of state prize of RK V.A. Kim (Zh. Abishev Chemical and Metallurgical Institute) [7].

Ash technical specifications and chemical composition of the carbonaceous reducers used in the experiments carried out by us are given in the Tables 1 and 2. Weight ratios of LGMC to the reducing agent were 1:0.25. Preparation of furnace charge for metallization was carried out by two ways, namely,

- LGMC and reducing agent (furnace coke or direksil) were placed in the melting pot by layers;
- LGMC and reducing agent (furnace coke or direksil) were carefully mixed and placed in the melting pot.

Table 1

Technical specifications of carbonaceous reducing agents

Reducing agent	Technical structure, %				
	W^p	A	V^d	S^d	P^d
Direksil	1.0–2.0	1.0–5.0	3.6–15.0	0.20–0.40	0.01–0.04
Furnace coke of ArselorMittal Temirtau	2.0–15.0	13.8	1.5	0.50	0.045

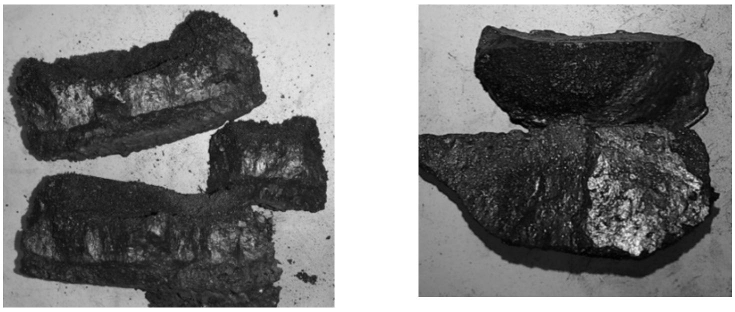
Table 2

Chemical composition of reducing agents' ash

Reducing agent	Content, %			
	SiO_2	Al_2O_3	MgO+CaO	Fe_2O_3
Direksil	48.06	18.62	4.78	7.32
Furnace coke of ArselorMittal Temirtau	48.50	20.0	8.7	16.6

The analysis of experiment results on metallization of LGMC with the use of furnace coke shows that at long isothermal holding the set temperature of emergence of the liquid phase is 1150 °C. In such a way it was observed emergence of the liquid phase — fusion (fayalite) in restoration products in the experiments carried out at the temperature of 1150 °C. Pictures of the metallized samples obtained as a result of reducing roasting of LGMC with the use of the furnace coke as a reducing agent at long isothermal holding of 60 and 90 minutes at 1150 °C are given in the Figure 1. As you can see on the fracture of the samples the fusion zone with increase in hold time up to 90 minutes covers almost total volume of the metallized product (sample *b*).

With reduction of isothermal time of LGMC mixture and solid reducing agent the temperature range of solid-phase restoration extends towards the greater temperature considerably. So at the time of isothermal holding of pilot samples within 5–15 minutes the noticeable emergence of the liquid phase was not observed at 1150 °C and even at 1200 °C. Noted feature of mixture behavior of LGMC and solid reducing agent is indicative of the basic possibility of realization of solid-phase restoration of LGMC at short-term holding of furnace charge within 10–15 minutes with achievement of the set extent of metallization of ≤40 % as well as ≥80 %. At the same time the subsequent separation of a ferriferous part from dead rock can be carried out by methods of magnetic separation of products of solid-phase metallization.



a — sample obtained at isothermal holding of 60 min

b — 90 min

Figure 1. Metallized samples obtained at reducing roasting of LGMC using furnace coke

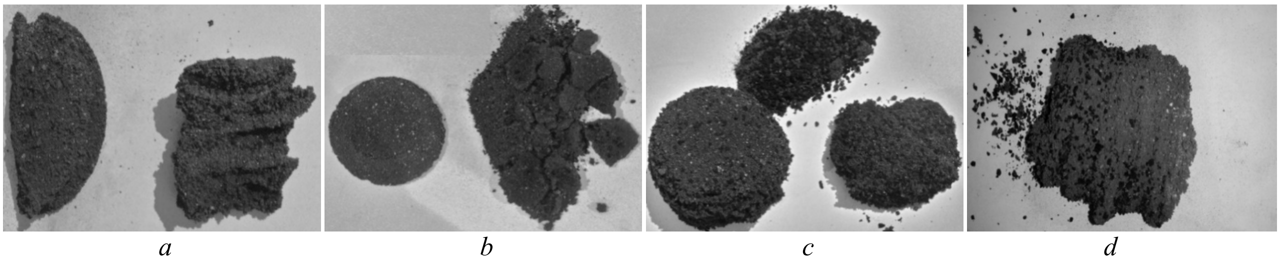
The concentrate which is formed at metallization extent of ≤40 % containing up to 72 % of iron can be used at domain melting of conversion iron. It opens a prospect of the wider involvement of brown iron ores into traditional two-stage steel production the balance reserves of which in Kazakhstan are estimated in billions of tons.

According to requirements to a metallized concentrate the criteria of its suitability as a furnace charge component for steel smelting in electric furnaces is metallization extent of more than 80 % at the minimum content of dead rock [8]. Results of the experiments carried out in the range of temperatures of 1150–1250 ºC showed that it was necessary to apply the highly reactive reducing agent in the form of direksil for achievement of metallization extent of LGMC of ≥80 %. At that in the temperature range of 1200–1250 ºC the set metallization extent of LGMC (80 % and more) is provided at mixture holding within 8–10 minutes.

In compliance with a research problem at traditional ways of metallization of LGMC connected with long isothermal holding, as a rule, exceeding 60–90 minutes there were set the technological parameters of solid-phase restoration in which metallization temperature should not exceed 1100 ºC. For obvious demonstration of influence of reducing agent type on metallization process of LGMC the results of the experiments carried out at 1100 ºC and hold time of 90 minutes are given below. The furnace coke and direksil are considered as the solid reducing agents (see Table 1). Conditions of furnace charge preparation are described below:

- Experiment No. 1 — LGMC and furnace coke were divided into 5 parts and placed in the melting pot by layers.
- Experiment No. 2 — LGMC and direksil were divided into 5 parts and placed in the melting pot by layers.
- Experiment No. 3 — LGMC and direksil were mixed and placed in the melting pot.
- Experiment No. 4 — LGMC and furnace coke were mixed and placed in the melting pot.

The pictures of the metallized product samples of LGMC obtainedas a result of solid-phase reduction with different reducing agents are given in the Figure 2.



a, b — furnace-charge by layers; *c, d* — mixed furnace-charge

Figure 2. Pictures of metallized product samples of LGMC

Products of reducing roasting are represented by weakly caked-on particles, which crumble by easy pressing. Thus, the physical condition of the metallization products obtained in the mode of solid-phase restoration allows using all known methods of separation of the metallized concentrate from the dead rock. And the most preferable option of such separation includes methods of magnetic separation in view of contrast formed after the system metallization. Separation of magnetic and non-magnetic products after metallization is an obligatory element of the technological scheme of LGMC metallization since the total content of iron in initial concentrate does not exceed 49 %. At that depending on metallization extent the magnetic fraction can be used as a furnace charge component when smelting the conversion iron in the blast furnace (metallization extent is less than 40 %) or steelmaking in the electric furnace (metallization extent is more than 80 %).

Samples *a* and *b* were obtained when loading furnace charge by layers, and samples *c* and *d* — as a result of carrying out metallization by furnace charge mixing. Direksil was used as a reducing agent in samples *b, d*; *a* and *c* were obtained with the use of furnace coke. For assessment of reduction degree of iron oxides on height of the metallized sample the test for the chemical analysis was selected by layers. Non-magnetic component of the metallized samples was separated by means of magnetic separation without crushing of the products obtained. The chemical composition of the magnetic part of the metallization products of LGMC is given in the Table 3. According to the results of analysis of the chemical composition of the magnetic component of metallized LGMC content of the total iron in the metallized products with application of direksil is higher than in samples with use of furnace coke for 5–7 abs. %.

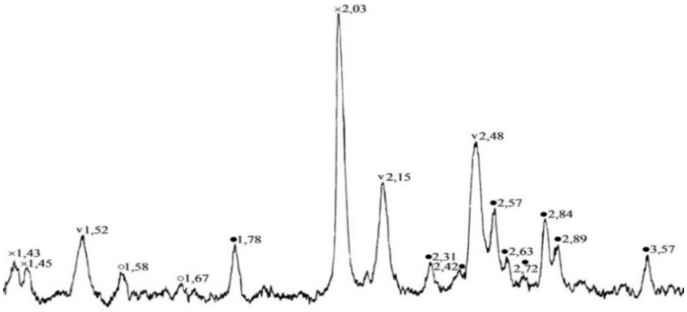
Table 3

Chemical composition of metallized products

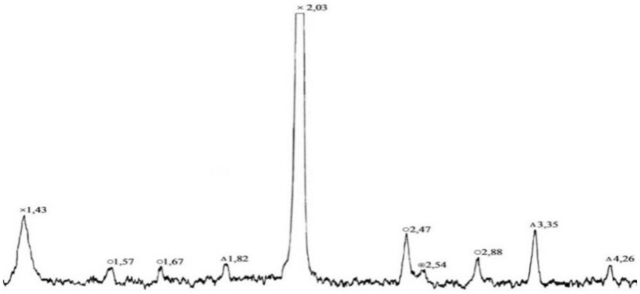
Exp. No.	Name	Content, %						
		Fe_{total}	SiO_2	Al_2O_3	CaO	MgO	P	S
1	LGMC with furnace coke by layers							
	1 (upper)	67.01	10.74	6.22	n/o	0.72	0.92	0.137
	2	67.40	10.81	6.18	0.87	n/o	0.98	0.134
	3	67.66	10.18	6.18	0.35	n/o	0.96	0.077
	4	67.66	8.99	6.37	0.52	n/o	0.99	0.077
2	LGMC with direksil by layers							
	1 (upper)	68.59	14.41	6.06	n/o	0.65	0.99	0.067
	2	77.22	10.50	7.04	0.52	n/o /o	1.1	0.057
	3	76.43	11.50	6.72	0.52	2.90	1.1	0.057
	4	76.69	11.22	7.19	n/o	2.90	1.1	0.057
3	LGMC with furnace coke by mixing							
	1 (upper)	71.26	7.84	6.40	n/o	0.99	0.97	0.173
	2	71.52	7.78	6.33	n/o	0.82	1.0	0.105
	3	70.32	8.04	6.29	n/o	0.82	1.0	0.115
	4	68.86	8.51	6.16	n/o	0.82	0.94	0.192
4	LGMC with direksil by mixing							
	1 (upper)	80.31	7.44	6.85	n/o	0.82	1.12	0.077
	2	77.91	8.31	6.68	n/o	0.98	1.08	0.096
	3	72.59	8.70	6.61	n/o	0.65	1.02	0.077
	4	70.72	8.31	6.29	n/o	0.82	0.99	0.096

In Figures 3 and 4 there are given diffraction patterns (X-ray patterns) of the metallized samples of LGMC characterizing phase structure and crystal structure of the obtained restoration products depending on the type of reducing agent. It should be noted that the content of silicon dioxide in the samples obtained by mixing the furnace charge before metallization process conduction is less for 3–5 % than in the samples obtained when loading furnace charge by layers that is coordinated with data of X-ray phase analysis (Fig. 3, 4).

As a result of solid-phase restoration in all experiments we have obtained a metallized concentrate in the form of weakly caked-on particles, which crumble by easy pressing. Extent of metallization is calculated according to the chemical analysis taking into account results of X-ray phase analysis. Exponents of metallization of the obtained products depending on conditions of furnace charge preparation for recovery roasting and the applied reducer are given in the Table 4.

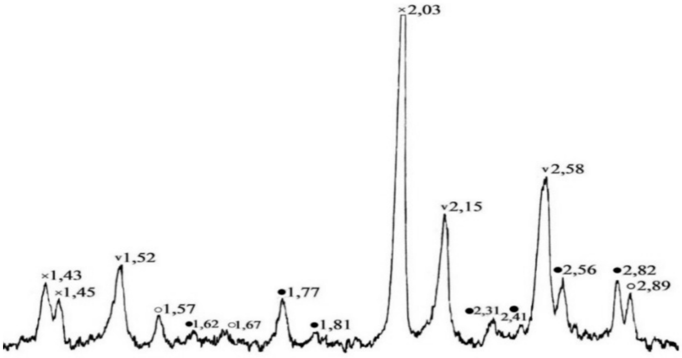


a — with furnace coke (x — metallic iron; V — wustite; ○ — hercynite; ● — fayalite)

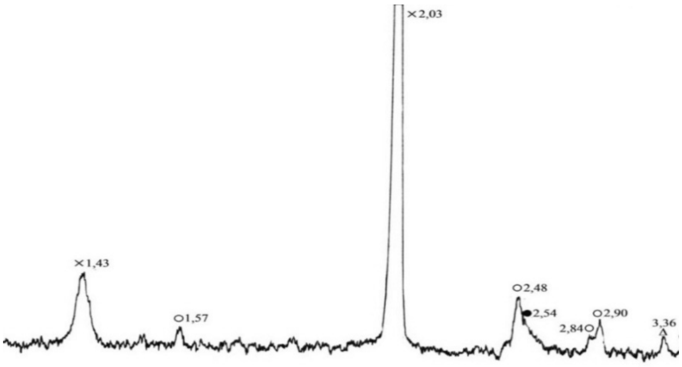


b — with direksil (x — metallic iron; Δ — quartz; ○ — hercynite; ʘ– magnetic iron)

Figure 3. Diffraction pattern of the metallized product samples obtained when loading the furnace charge by layers with application of furnace coke (*a*) and direksil (*b*) as a reducing agent



a — with furnace coke (× — metallic iron; V — wustite; Δ — quartz; ○ — hercynite; ● — fayalite)



b — with direksil (× — metallic iron; Δ — quartz; ○ — hercynite; ● — fayalite)

Figure 4. Diffraction pattern of the metallized product samples obtained when mixing the furnace charge with application of furnace coke (*a*) and direksil (*b*) as a reducing agent

Table 4

Metallization degree of roasted product of LGMC

Exp. No.	Name	T, °C	τ, min	Fe_{total}, %	Fe_{met}, %	α_{met}, %
1	LGMC with furnace coke by layers	1100	90			
	1 (upper)			67.01	17.13	25.26
	2			67.40	47.74	29.19
	3			67.66	16.60	24.53
	4			67.66	13.77	20.35
2	LGMC with direksil by layers	–"–	–"–			
	1(upper)			68.59	36.03	52.53
	2			77.22	67.55	87.47
	3			76.43	67.75	88.64
	4			76.69	67.78	88.40
3	LGMC with furnace coke by mixing	–"–	–"–			
	1(upper)			71.76	27.98	39.26
	2			71.52	27.95	39.07
	3			70.32	23.31	33.15
	4			68.86	18.05	26.21
4	LGMC with direksil by mixing	–"–	–"–			
	1(upper)			80.31	68.41	85.18
	2			77.91	66.68	85.50
	3			72.59	36.39	50.13
	4			70.72	34.32	48.52

According to the chemical analysis (Table 4) the greatest extent of metallization of 85–88 % at the total iron content of 72–77 % was reached in the experiments No. 2 and No. 4, when using direksil as a reducing agent, activity of which is much higher than those of traditionally applied reducers, in particular, of furnace coke. At that the metallization extent in the experiment No. 2 (with direksil) is 3 times higher than in the experiment No. 1 (with furnace coke). Diffraction patterns of the metallized productsamples given in Figures 3 and 4 also confirm advantage of using direksil as a reducing agent. Characteristic peaks of metal iron are much more intensive, and presence of fayalite and hercynite, which are formed in the course of the iron oxides recovery, is insignificant (Fig. 3, *b* and 4, *b*) in comparison with phase structure of the metallized product obtained with the use of furnace coke (Fig. 3, *a* and 4, *a*). The average chemical composition of the metallized samples is as follows Fe_{total} — 77.91; SiO_2 — 8.31; $A1_2O_3$ — 6.68; MgO — 0.98; P — 1.08. Basicity $(A1_2O_3+MgO)/SiO_2$ is equal to 0.9.

Thus, as a result of conducting studies of the metallization modes of brown iron lisakovsky concentrate there are set the rational parameters of direct solid-phase restoration of LGMC in the laboratory mine furnace providing extent of its metallization of 20.38–88.64 %. It is more preferable to usedireksilas a reducing agent, highly active solid reducer from Shubarkol coal allowing raising extent of metallization of LGMC in more than 3 times in comparison with furnace coke. Optimum temperature of solid-phase metallization process of LGMC at long holding should not exceed 1100 ºC. With reduction of time of isothermal holding of LGMC mixture with solid reducer the solid-phase metallization is provided at more high temperatures of roasting reaching up to 1150–1200 ºC. The specified feature allows using the new technical solutions for achievement of the set metallization extents of LGMC obtaining the final products for multiple purposes.

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Қоңыртемірлі лисаков шикізатының қатты фазалық металдану үрдісін зерттеу

«Дирексил» жоғары реакционды қатты көміртекті тотықсыздандырғышты қолдану арқылы лисаков гравитациялық магнитті концентратын (ЛГМК) металдандыру үрдісіне зерттеу жүргізілді. 20,4–88,6 металдану дәрежесін қамтамасыз ететін ЛГМК-ның қатты фазалық тотықсыздану үрдісінің ұтымды көрсеткіштері орнатылды. Жоғары жылдамдық пен темір тотығының металдану деңгейін қамтамасыз етіп, сұйық фаза — балқыманың қалыптасуынан айналып өтетін қатты фазалы темірдің тікелей қалыптасуының 1150–1200 ºС-дағы температура диапазоны анықталды. «Дирексилды» тотықсыздандырғыш ретінде пайдалану ЛГМК-ның металдану деңгейін домна коксымен салыстырғанда 3 еседен көп арттыруға мүмкіндік береді. Қалыпқа келтірушінің түріне байланысты алынған қалыпқа келтіруші өнімдердің кристалдық құрылымы мен фазалық құрамын сипаттайтын металданған ЛГМК түрлерінің дифрактограммалары (рентгенограммалары) келтірілген. Қатты фазалық қалыпқа келтіру тәртібінде алынған металданған өнімдердің физикалық күйі бос түрлерден металданған концентратты бөлуде барлық белгілі әдістерді пайдалануға мүмкіндік береді. Жүйелердің металдануынан кейін қалыптасатын қарама-қайшылығындай магнитті сепарация амалдары мұндай бөлулерде ең қолайлы нұсқа болып табылады. Металдандырудан кейін өнімдерді магнитті және магнитсіз бөлу ЛГМК металдандырудың технологиялық сызбасының міндетті элементі болып табылады, себебі шығыс концентратында жалпы темір құрамы 49 % аспайды. Сонымен қатар металдандыру деңгейіне байланысты магнитті фракция домендi пеште қолданбалы шойынды қорытудағы (металдандыру деңгейі 40 %-дан кем емес) немесе электр пеште болатты балқытудағы (металдандыру деңгейі 80 %-дай) шихта компоненті сияқты пайдаланылуы мүмкін.

Кілт сөздер: қоңыр темір кендері, ЛГМК, қатты фазалық металдандыру, металдандыру деңгейі, көміртекті тотықсыздандырғыш, «Дирексил», фазалық құрамы, металдандырылған концентрат.

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Исследование процесса твердофазной металлизации бурожелезнякового лисаковского сырья

В статье приведены результаты исследования процесса твердофазной металлизации лисаковского гравитационного магнитного концентрата (ЛГМК) с применением высокореакционного твердого углеродного восстановителя «Дирексил». Установлены рациональные параметры процесса твердофазного восстановления ЛГМК в шахтной печи, обеспечивающие степень его металлизации 20,4–88,6 %. Определен температурный диапазон твердофазного прямого восстановления железа 1150–1200 ºС, обеспечивающий максимальную скорость и степень металлизации оксидов железа, минуя при этом появление жидкой фазы — расплава. Использование «Дирексила» в качестве восстановителя позволяет повысить степень металлизации ЛГМК в более чем 3 раза по сравнению с доменным коксом. Приведены дифрактограммы (рентгенограммы) металлизованных образцов ЛГМК, характеризующие фазовый состав и кристаллическую структуру полученных продуктов восстановления в зависимости от вида восстановителя. Физическое состояние продуктов металлизации, полученных в режиме твердофазного восстановления, позволяет использовать все известные методы разделения металлизованного концентрата от пустой породы. Причем наиболее предпочтительным вариантом подобного разделения являются приемы магнитной сепарации в виду контрастности образующейся после металлизации системы. Разделение магнитного и немагнитного продуктов после металлизации является обязательным элементом технологической схемы металлизации ЛГМК, так как суммарное содержание железа в исходном концентрате не превышает 49 %. При этом, в зависимости от степени металлизации, магнитная фракция может быть использована как компонент шихты при выплавке передельного чугуна в доменной печи (степень металлизации менее 40 %) или же выплавке стали в электрической печи (степень металлизации более 80 %).

Ключевые слова: бурожелезняковые руды, ЛГМК, твердофазная металлизация, степень металлизации, углеродный восстановитель, «Дирексил», фазовый состав, металлизованный концентрат.

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