Досліджено фізико-хімічні властивості продуктів вуглецевотермічного відновлення оксидної хромвмісної рудної сировини. Це необхідно для визначення параметрів, що знижують втрати Cr при переробці рудних матеріалів і використанні металізованих хромвмісних легуючих добавок у сталеплавильному виробництві. Визначено, що підвищення температури обробки з 1250 K до 1450 K призвело до збільшення прояву $Cr_{23}C_6$ та $(Cr, Fe)_7C_3$. При цьому дифракційні максимуми Cr_2O_3 відповідали тенденції послаблення та після обробки при 1450 K мали залишковий характер. Cr_3C_2 на дифрактограмах проявлявся лише після обробки при 1250 K. Фаза металевого Cr прослідковувалась в зразках після обробки при 1350 K та 1450 K з підсиленням інтенсивності прояву при збільшенні температури нагрівання. Визначено, що мікроструктура продуктів відновлення неоднорідна з присутністю часток різного розміру та хімічного складу. З підвищенням температури відновлення з 1250 K до 1350 K та 1450 K і розвитком відновних процесів мало місце спікання часток з утворенням губчастої мікроструктури. Виявлено ділянки, що характеризували включення і фази із вмістом Cr до 65,10 % мас., Fe – до 16,13 % мас. Також виявлено окремі локальні ділянки з частками з відносно високим вмістом рудних домішок та вуглецю. З отриманих результатів дослідження витікає, що найбільш прийнятною температурою для відновлення є 1450 K. В цьому випадку забезпечується відновлення з переважанням у фазовому складі Cr металевого та карбідів $(Fe, Cr)_7C_3$ і $Cr_{23}C_6$ відносно оксидної складової Cr_2O_3. При цьому менший залишковий вміст вуглецю обумовлений більш високою ефективністю дії відновника порівняно з іншими температурними режимами. Завдяки губчастій мікроструктурі стає можливим більш швидке розчинення, порівняно із стандартними феросплавами, при цьому реалізується скорочення часу виплавки

Ключові слова: хромвмісна оксидна рудна сировина, фазовий аналіз, вуглецевотермія, карбід, легування, відновлення

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REVEALING NEW PATTERNS IN RESOURCE-SAVING PROCESSING OF CHROMIUM-CONTAINING ORE RAW MATERIALS BY SOLID-PHASE REDUCTION

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1. Introduction

Current trends in the steel industry are aimed at increasing demand for steel doped with rare and refractory elements. One such element is Cr. Gradual depletion of raw materials deposits leads to a growing price in the world market for refractory doping materials [1]. Traditional technologies for obtaining Cr-based doping materials using carbon-sili-

cothermic and aluminothermic melting are characterized by significant temperatures and processing times. Accordingly, these processes are accompanied by relatively high pollution of the environment by gaseous products of reactions and solid waste [2], as well as significant resource and energy costs.

The main indicators determining the competitiveness of metal products in the domestic and global market are their quality and cost [3]. Resource conservation issues are most relevant for the special metallurgy where expensive doping additives are used. In part, these additives, due to specific physical and chemical properties, pass into slag, sludge, scale [4], and other metallurgical waste.

The technologies used to produce ferroalloys based on refractory elements, including Cr, have almost exhausted their reserves in terms of improving the efficiency and quality of doping materials. The technologies are characterized by high processing costs and irretrievable losses of the target element with waste [5].

During steel smelting, the long-term aging of the melt to dissolve the ferroalloys and the averaging of the chemical composition reduces the performance of the furnace units. Reducing steel production costs can be achieved by replacing some of the used standard chromium ferroalloys with spongy doping materials with qualitatively new, more beneficial properties [6].

Modern technologies for obtaining refractory materials by powder metallurgy methods partially solve existing problems [7]. However, the reserves of improving resource-saving technologies for obtaining doping materials based on chromium ore concentrates with specified physical and chemical properties for the special metallurgy are far from exhausted.

Therefore, it is a relevant task to save resources in steelmaking, together with increasing the degree of Cr absorption during processing and the use of chromium-containing ore materials. A significant direction in addressing this issue is to develop ideas about the mechanism of reduction of oxide chromium-containing raw materials and to determine the physical and chemical properties of the resulting products.

2. Literature review and problem statement

The use of powder metallurgy makes it possible to obtain products with qualitatively new consumer properties and opens the possibility of further improvement [8]. Carbon thermal treatment is one of the technologically simple and economically attractive ways of solid-phase reduction when processing by the methods of powder metallurgy the oxide materials that contain iron and refractory elements [9]. According to [10], Fe_3C and C were detected in the reduction products following the carbon thermal treatment of iron-containing oxide raw materials. Similar results were reported by authors of work [11] when reducing by carbon thermal treatment technogenic iron-chromium-containing oxide raw materials. Based on the results of phase analysis, doping elements are present not as separate compounds but as a solid solution in the Fe phase. The carbon reduction of highly doped refractory elements, including Cr, of technogenic oxide raw materials was studied by authors of [12]. It was established for the products with varying degrees of reduction that most of the doping elements were in the carbide iron compounds and the solid solution of α-Fe as replacement atoms. Only when the reduction degree reached 62 % after heat treatment at 1,523 K, the phase composition of reduction products revealed the iron-chromium-containing carbide $(Fe, Cr)_7C_3$. That is, the presence of doping elements in the carbon-thermally reduced products was manifested to a larger degree by the oxide complex with iron atoms. This should be taken into consideration in further research into the reduction and processing of ore raw materials. However, there is a need to deepen the understanding of the physical and chemical patterns of interaction between chromium-containing ore raw materials and the reducer.

The importance of carbon monoxide decomposition in the regenerative processes in the Fe–Cr–O–C system was noted in work [13]. In the reactions between Cr_2O_3 and C, the first stage is the dissociation of oxides with the release of atomic and molecular oxygen and the formation of carbon oxides. Further reduction of Cr is provided by the reaction between Cr_2O_3 and the C_3O_2 oxide compound and C, which are formed from the decomposition of carbon oxides. That is, it is necessary to emphasize the important function of the gas phase during carbon-thermal reduction and take this into consideration when developing appropriate technological parameters. However, the results of the cited work do not give an idea about the effect of ore impurities on the patterns of the reduction of oxide chromium-containing raw materials.

The authors of work [14] experimentally investigated the reduction of chromium-containing ore raw materials by SiC particles in the temperature range 1,389–1,661 K. They identified the formation of metal particles with components of Fe, Cr, and Si, and noted the need for temperatures above 1,473 K during reduction. However, the temperatures for the equilibrium shift towards reduction reactions involving SiC would be different when using pure carbon reducers.

Restoring the $FeO{\cdot}Cr_2O_3$ oxides at various C:Fe ratios and temperatures from 1,373 K to 1,523 K was studied by authors of work [15]. It was determined that with the increase in C:Fe from 0.8 to 1.4 the degree of retrieval (%) of Cr increased from 9.6 to 74.3, respectively. Increasing the temperature to 1,523 K led to an increase in the formation of carbides, some of which are inevitably present in the carbon-thermally reduced products [16]. At C:Fe below 0.8, there was a significant decrease in the Cr extraction and a decrease in carbide formation. The formed Cr carbides dissolved in the Fe phase [15]. The formation of the metal Cr and Cr carbides was also discovered by authors of paper [7] in the reduction of chromium-containing ore raw materials. After heat treatment at 1,273 K and O:C in the charge between 1.05 and 1.15, the carbon thermal reduction was accompanied by the evolution of Cr_3C_2 and Cr_7C_3. That is, it turns out that to increase the degree of Cr extraction during reduction, the composition of the charge should contain some excess of carbon relative to oxygen. Studies [17, 18] reported the results of reducing Cr_2O_3 by carbon in the range 1,273–1,773 K. The parallel reduction and formation of carbides Cr_3C_2, Cr_7C_3, $Cr_{23}C_6$ was found to be taking place; at the same time, the possibility of obtaining iron-chromium ligatures with a limited carbon content was established. It is possible to obtain the carbon-thermally reduced products with relatively low residual carbon, which indicates an extended scope of application. However, it is not possible to trace the patterns of change in the phase composition and microstructure of the received materials when reducing the complex of Cr and Fe oxides in the composition of ore raw materials at different temperatures.

One should note significant results of studying the carbon-thermal reduction of oxide iron-containing [10] and doped raw materials with Cr content [11, 12]. The possibility to form a solid solution of carbon and doping elements in α-Fe and Fe_3C and $(Fe, Cr)_7C_3$ carbides was established. Also important are the results reported in work [13] involving the decomposition of carbon oxides during reduction processes in the Fe–Cr–O–C system. But at the same time, there are the disregarded notions about the influence of ore impurities on the patterns of the reduction of oxide chromium-containing raw materials.

Research into the reduction of chromium-containing ore raw materials involving SiC, described in paper [14], expands the range of use of possible reducers, including abrasive waste. However, the cited results do not give an answer regarding the parameters of the reduction of chromium-containing ore raw materials by purely carbon reducers.

A study of the carbon thermal treatment of oxides in the Fe–Cr–O–C system [15] deepens the understanding of the reduction of the oxide component of chromium-containing ore raw materials. In this case, rather important are the results reported in work [7] that confirm the processes of the formation of metallic Cr, Cr_3C_2, and Cr_7C_3. Along with this, $Cr_{23}C_6$ can form [17] with the possibility of obtaining iron-chromium ligatures with a limited carbon content [18]. However, the issue of the impact of reduction factors, including the temperature characteristics of the process, on the phase and structural component of the target products is not studied in detail. Research in this field can reduce Cr losses when receiving and using the reduced doping material.

Therefore, it is appropriate to study the patterns of the effect of the temperature of the reduction of chromium-containing ore raw materials on the phase composition and microstructure of the obtained doping material. This could define the character of the presence of elements in reduction products. In this case, the application of X-ray microanalysis of individual inclusions and phases would enhance the understanding of the distribution of doping elements in the resulting doping additive. This is necessary to determine the parameters that reduce the loss of Cr in the processing of ore materials and the use of the metallized chromium-containing doping additives in steelmaking. The research in this field is also needed to further advance the ideas about the mechanism of reduction and carbide-forming processes in the metallization of oxide chromium-containing materials.

3. The aim and objectives of the study

The aim of this study was to identify new patterns of resource-saving processing of chromium-containing ore raw materials by the solid-phase reduction.

To accomplish the aim, the following tasks have been set:

- to determine the features of the phase composition of the carbon-thermally reduced products from chromium-containing ore raw materials at different processing temperatures relative to the effect on reducing the loss of the target element during doping;
- to investigate the microstructure and chemical composition of individual phases and inclusions of the carbon-thermally reduced products from chromium-containing ore raw materials at different thermal treatment regimes regarding the nature of the presence of the elements.

4. Materials and methods to study the properties of the carbon-thermally reduced products from chromium-containing ore raw materials

4. 1. Examined materials, and equipment used in the experiment

The main component of the charge is industrial finely-dispersed chromium ore (TU 14-9-220-81). The grinding was performed at a ball mill with peripheral unloading of the ground material. Sifting was carried out through a sieve with a cell size of 0.45 mm. The reducer is carbon, in the form of ultra-dispersive dust from carbon production (carbon share is 98 % by weight), the addition of which provided a ratio of O:C in the charge at 1.33. The reduction was carried out in the furnace of indirect heating with a coal lining. The temperature interval of heat treatment is 1,250–1,450 K; the isothermal aging time is 60 minutes. The protective environment is the atmosphere of argon with a linear gas flow rate of 2.5 and 10^{-3} m/s. Mass of the examined samples before heat treatment is 80 grams.

X-ray analysis of the samples was performed at the "DRON-6" (Russia) diffractometer.

Photographs of the microstructure and chemical composition of individual sections of the sample surface were acquired from the JSM 6360LA electron microscope, equipped with the JED 2200 X-ray microanalysis system, manufactured by JEOL (Japan).

4. 2. Procedure of determining the samples' properties indicators

The phase composition of the examined samples was determined by X-ray analysis using the monochromatic radiation of Cu Kα (λ=1.54051 Å). Measurements were performed at a voltage on the tube of U=40 kV and anode current I=20 mA. The composition of the phases was determined by employing the PDWin 2.0 (Russia) software suite.

The microstructure of the samples was examined at an accelerating voltage of 10–25 kV and an electron probe current 52–96 μA. The working distance to the examined surface was 10.5–11.7 mm.

The composition of the phases was determined by a reference-free method of calculating fundamental parameters: calculating the correcting ratios of reflection of the probe's electrons, absorption of characteristic X-ray radiation, and fluorescence. We determined the chemical composition of the phases on the sections marked in the photographs of the microstructure by the corresponding designations.

5. Results of studying the properties of carbon-thermally reduced products from chromium-containing ore raw materials

5. 1. Results of studying phase composition

After heat treatment at 1,250 K, the phase composition of reduction products is represented by Cr_2O_3 and carbides Cr_3C_2, $Cr_{23}C_6$ and $(Cr, Fe)_7C_3$ (Fig. 1).

The increase in temperature to 1,350 K reduced the intensity of Cr_2O_3 manifestation. In this case, there was a disappearance of the Cr_3C_2 diffraction maxima. At the same time, the presence of the metallic Cr phase was detected in the reduction products and the intensity of the manifestation of $Cr_{23}C_6$ and $(Cr, Fe)_7C_3$ increased. After processing at 1,450 K, the $Cr_{23}C_6$ and $(Cr, Fe)_7C_3$ carbides demonstrated

the highest intensity of diffraction maxima in the reduced products from chromium-containing ore raw materials. In this case, there was an increase in the manifestation of the phase of metallic Cr. The presence of the Cr_2O_3 oxide component was residual and was characterized by a relatively weak manifestation.

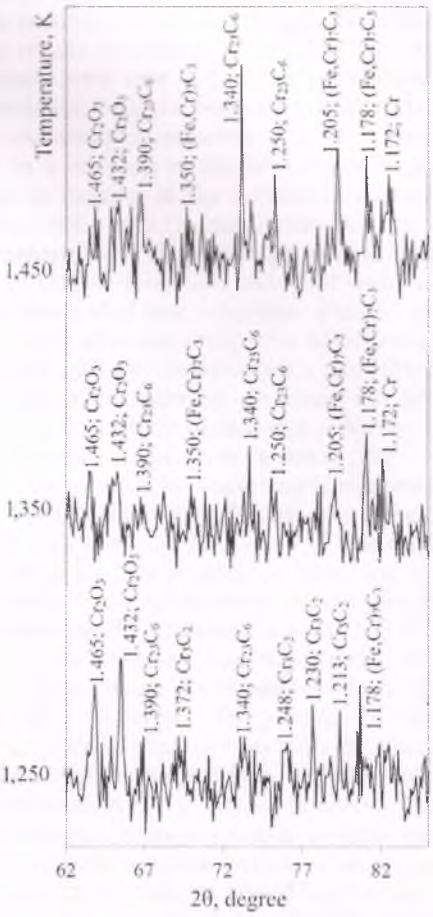

Fig. 1. X-ray phase study of the reduced products from chromium-containing ore raw materials after processing at different temperatures

5. 2. Results of studying the microstructure and chemical composition of individual phases and inclusions

The microstructure of the samples is heterogeneous with the presence of particles of different sizes (Fig. 2) and chemical composition (Table 1, Fig. 3).

The development of reduction processes was accompanied by particle sintering with the formation of a spongy microstructure. We identified sections with Cr content up to 65.10 % by weight, Fe – up to 16.13 % by weight. We also detected sections with the relatively high content (% by weight) of ore impurities: Mg, Al, and Si – 33.51, 4.55, and 16.64, respectively. Some of the examined sections were characterized by a relatively high content of carbon and oxygen – 43.6 % by weight and 39.57 % by weight, respectively.

Table 1

Results of the X-ray microanalysis of samples of the reduced chromium-containing ore raw materials corresponding to Fig. 2

Section	Element content, % by weight							
	C	O	Mg	Al	Si	Cr	Fe	Total
1	9.60	14.23	7.35	4.55	0.00	50.71	13.56	100.00
2	6.46	10.04	7.51	1.57	0.26	60.56	13.60	100.00
3	3.77	7.84	4.63	2.17	0.36	65.10	16.13	100.00
4	43.60	9.12	7.75	0.93	7.64	22.27	8.69	100.00
5	5.16	39.57	33.51	0.00	16.64	0.00	5.12	100.00

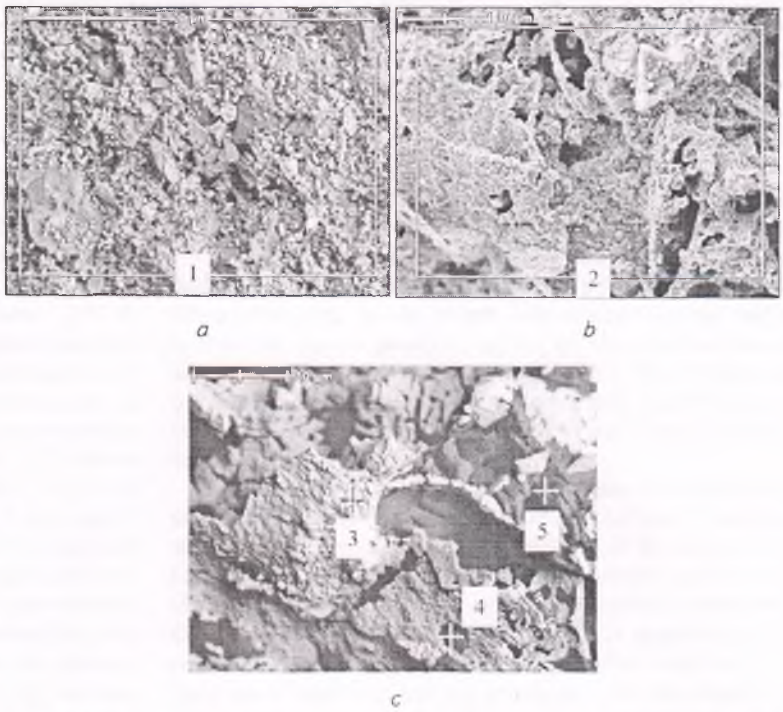

Fig. 2. Microstructure of the reduced chromium-containing ore raw materials corresponding to Fig. 1 after varying processing temperatures with varying degrees of magnification: *a* – 1,250 K, ×500; *b* – 1,350 K, ×1,000; *c* – 1,450 K, ×1,000, 1–5 – sections of the X-ray microanalysis of samples

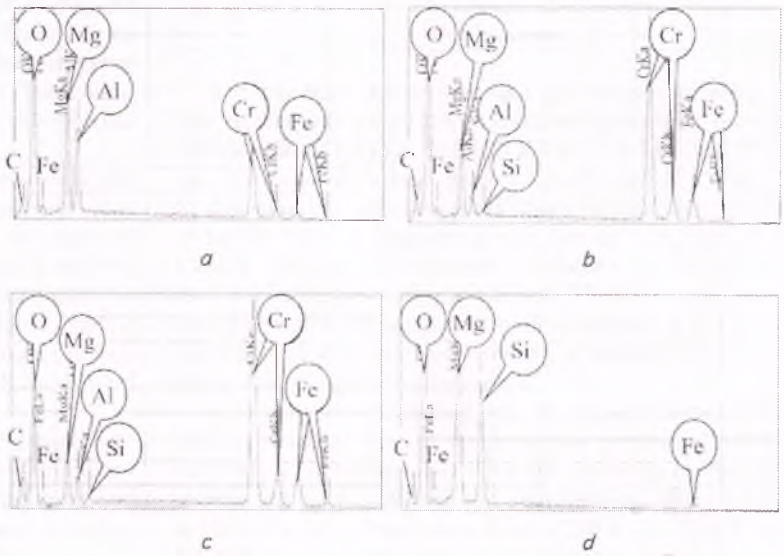

Fig. 3. Spectrograms of some examined sections corresponding to Fig. 2: *a* – 1, *b* – 2, *c* – 3, *d* – 5

6. Discussion of results of studying the indicators of properties of the carbon-thermally reduced products from chromium-containing ore raw materials

It has been determined that treatment at 1,250 K did not provide a sufficient level of reduction with the Cr_2O_3 dominating the phase composition (Fig. 1). In this case, the presence of Cr_3C_2 and $Cr_{23}C_6$ carbides agrees well with the results reported in [7, 17]. And the detection of double carbide with iron and chromium content $(Fe, Cr)_7C_3$ is consistent with the results reported in [12]. The increase in processing temperature to 1,350 K and 1,450 K resulted in a decrease in the intensity of Cr_2O_3 manifestation and an increase in the diffraction maxima of $Cr_{23}C_6$ and $(Fe, Cr)_7C_3$ with the manifestation of metallic Cr. This is consistent with the results given in [15] when the intensity of carbide formation increased with an increase in the temperature of heat treatment. Parallel carbide formation is clearly observed along with metallization, which is consistent with the results reported in [10]. Parallel carbide formation indicates an extremely low probability of obtaining completely carbon-free reduction products, which corresponds to the results given in [16].

The increase in processing temperature from 1,250 K to 1,350 K and 1,450 K leads to a more explicit particle sintering with the formation of a spongy microstructure (Fig. 2). The progress of reduction is evidenced by a decrease in the residual content of C and O in the reaction products at the examined sections 1–3 (Table 1). In these areas, the C content decreased from 9.60 % by weight to 3.77 % by weight, the O content – from 14.23 % by weight to 7.84 % by weight. The presence of residual C together with Cr and Fe agrees well with the phase study and further indicates the presence of the carbide or oxy-carbide component in the reduction products. The intensification of reduction processes is also possible due to the spongy microstructure, which facilitates the access of the gaseous reducer to the reaction area. This is in line with the results reported in [13], which describes the mechanism of formation and the significant role of the gas-forming reduction component in the Fe–Cr–O–C system.

Some local examined particles had a relatively high content of Mg, Si, and O (% by weight) – 33.51, 16.64, and 39.57 respectively (Table 1, Fig. 3, section 5). These can be particles of oxide impurities that enter the reduction products along with ore. That is, it is possible that these impurities may have an indirect effect on the course of reduction and carbide formation processes.

Section 4 in Fig. 2 with a relatively high carbon content of 43.60 % by weight can point to the residual local particles of an unused carbon reducer. The post-reduction of the oxide chromium-containing component would proceed directly when the received material is introduced into the molten metal as a doping additive. This is due to excess carbon from carbide and oxy-carbide compounds, as well as from the identified section of samples with an unused carbon reducer.

The absence of compounds with a relatively high propensity for sublimation in the resulting samples eliminates the need to create conditions that prevent evaporation and irretrievable losses with the gas phase of doping elements. This increases the reduction rate of doping elements.

It follows from the results of our study that the most acceptable temperature for reduction is 1,450 K. In this case, the reduction was ensured with the phase composition dominated by metallic Cr and carbides $(Fe, Cr)_7C_3$ and $Cr_{23}C_6$ relative to the Cr_3C_2 oxide component. At the same time, there was a more complete utilization of the carbon reducer, predetermined by lower residual carbon content compared to other temperature regimes.

A limitation worth noting is the disregard of the patterns of the effect of carbon content in the charge on the physical and chemical properties of reduction products from oxide chromium-containing ore raw materials. Research in this field could lead to the most appropriate conditions for extracting the target element from ore raw materials during reduction and when added as a doping additive. Such a study is therefore appropriate in the future.

The current study could be advanced by considering more types of ore oxide raw materials, which would be involved in processing by the solid-phase reduction technique. The difficulty in trying to advance this study is the lack of a sufficient database of experimental data. The most promising in this aspect are the ore concentrates of refractory elements.

Replacing carbon-silicothermic and aluminothermic melting with more efficient powder metallurgy methods when receiving a chromium-containing doping additive would have a positive impact on the environmental stresses experienced by industrial regions. This is due to a decrease in energy and resource costs and harmful emissions to the environment compared to traditional melting technologies.

The use of the obtained reduced chromium-containing material is feasible for doping instrumental steels with no strict carbon restrictions when smelting at an electric arc furnace. Pre-briquetting before heat treatment could make the material more compact and technologically advanced. Owing to the spongy microstructure, it is possible to dissolve faster than standard ferroalloys, while reducing the duration of smelting. And the improved reducing capability could provide for a higher degree of Cr assimilation. This, in turn, leads to a decrease in technogenic emissions and a decrease in the energy used, which results in the more environmentally friendly industrial conditions.

7. Conclusions

1. It has been determined that the increase in the processing temperature of oxide chromium-containing ore raw materials from 1,250 K to 1,450 K led to an increase in the manifestation of carbide phases $Cr_{23}C_6$ and $(Cr, Fe)_7C_3$. In this case, the diffraction maxima of Cr_2O_3 corresponded to the trend of weakening and, having been treated at 1,450 K, had a residual character. Carbide Cr_3C_2 manifested itself only after processing at 1,250 K. The Cr metallic phase was observed in the samples after processing at 1,350 K and 1,450 K with the increased intensity of manifestation when the heating temperature increased.

2. It has been established that the microstructure of the reduced products from oxide chromium-containing ore raw materials is heterogeneous with the presence of particles of different sizes and chemical compositions. The increase in the reduction temperature from 1,250 K to 1,350 K and 1,450 K and the development of reduction processes were accompanied by particle sintering with the formation of a spongy microstructure. We have identified sections that

characterized the inclusions and phases with Cr content up to 65.10 % by weight, Fe – up to 16.13 % by weight. We have also detected sections with particles with a relatively high content (% by weight) of ore impurities: Mg, Al, and Si – 33.51, 4.55, and 16.64, respectively. Some of the inclusions studied were characterized by a relatively high content of carbon and oxygen – 43.6 % by weight and 39.57 % by weight, respectively.

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