UDC 669.15'28-198
DOI: 10.15587/1729-4061.2017.108191

STUDY INTO PROPERTIES OF THE RESOURCE-SAVING CHROMIUM-CONTAINING BRIQUETTED ALLOYING ADDITIVE FROM ORE RAW MATERIALS

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Досліджено фазовий склад, мікроструктуру хромвмісної оксидної рудної сировини і відповідних продуктів вуглецевотермічного відновлення. Дослідно-промисловим шляхом отримано ресурсозберігаючий хромвмісний брикетований легуючий матеріал з якісно новими властивостями. Виявлено неоднорідну оксикарбідну структуру з надлишковим вмістом вуглецю, що забезпечує довідновлення залишкової оксидної складової при легуванні та захист від вторинного окислення хрому

Ключові слова: хромвмісна оксидна рудна сировина, вуглецевотермія, фазовий аналіз, мікроструктура, ресурсозбереження, легування

Исследованы фазовый состав, микроструктура хромсодержащего оксидного рудного сырья и соответствующих продуктов углеродотермического восстановления. Опытно-промышленным путем получен ресурсосберегающий хромсодержащий брикетированный легирующий материал с качественно новыми свойствами. Выявлена неоднородная оксикарбидная структура с избыточным содержанием углерода, что обеспечивает довосстановление остаточной оксидной составляющей при легировании и защиту от вторичного окисления хрома

Ключевые слова: хромсодержащее оксидное рудное сырье, углеродотермия, фазовый анализ, микроструктура, ресурсосбережение, легирование

1. Introduction

Current trends in metallurgy are aimed at increasing the demand for steel, alloyed by rare and refractory elements. One of such elements is Cr. Gradual depletion of raw material deposits predetermines a trend of rising prices in the world market on refractory alloying materials [1].

Comprehensive planning of metallurgical production is impossible without taking into account indicators of energy and material consumption of the finished product [2]. Metallurgy of rare and refractory metals and their respective alloying materials is characterized by considerable energy and material consumption, which increases the importance of this indicator.

The development and application of promising directions in powder metallurgy for receiving the alloying materials based on Cr gradually replaces traditional technologies of alumo- and carbon-silicon thermal melting [3]. This in turn leads to a reduction in harmful emissions into the atmosphere and, as a consequence, to a decrease in ecological stress. The use of powder metallurgy makes it possible to obtain products with qualitatively new consumer properties and opens up the possibility of further improvement [4].

Thus, it is a relevant task to save resources in steelmaking, along with an increase in the degree of the Cr application in the processing and use of the ore chromium-containing materials. Strategic direction in solving this problem is to develop an understanding about the mechanism of reducing the oxide chromium-containing raw materials. Along with this, essential task is the improvement of ecological situation in industrial regions. It is possible to achieve positive results in addressing this problem by replacing resource- and energy-intensive technologies of alumo- and carbon-silicon thermal melting with modern methods of powder metallurgy.

2. Literature review and problem statement

Article [5] reported an analysis of thermodynamic regularities in reducing Cr_2O_3 with carbon. The existence of physical-chemical prerequisites was noted for extracting Cr from the ore materials at relatively low temperatures without melting the charge. That is, the possibility is predetermined of replacing alumo- and carbon-silicon thermal melting [6] with the more effective resource- and energy-saving processes of solid-phase reduction.

Papers [7, 8] present theoretical and experimental research into carbon-thermal reduction of Cr_2O_3 in a temperature range of 1273–1773 K. A leading role was defined of CO and CO_2, which enable a bond between solid reagents. Given this, one should note the importance of providing a certain level of porosity after the briquetting of raw materials in order to implement a satisfactory gas exchange. Authors of articles [7, 8] also established a principal possibility to obtain iron-chromium ligatures with a limited content of carbon in the solid state under the energy-saving conditions of low temperatures. It demonstrates positive prerequisites for the development of research in the direction of receiving the resource- and energy-saving chromium-containing briquetted alloying additives with the assigned qualitatively new consumer properties.

The importance of decomposition of carbon oxides during reduction processes in the system Fe–Cr–O–C is noted in article [9]. In the interaction 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 of Cr_2O_3 with oxide compound C_3O_2 and C, which are formed from the decomposition of carbon oxides. In other words, one should emphasize an important function of the gas phase during carbon-thermal reduction and to take it into account during development of the respective technological parameters.

The use of carbon reducing agents inevitably leads to the presence of residual carbon in the received products bound in the oxy-carbide and carbide compounds [10, 11]. This should be especially considered when using the derived reduced materials for alloying the steels and alloys with strict limits on carbon. One of the effective carbon reducing agents is carbon black. Amorphous finely dispersed carbon black structure contributes to the activation of kinetics of reduction processes [12]. The specified properties make it possible to achieve a relative reduction in the time of reduction, and, therefore, to bring down the cost of technological energy. When using this type of a reducing agent, articles [13, 14] experimentally confirmed the diffusion character of carbide formation in the system Cr–C. It was found that in the mixture with a Cr/C ratio equal to 3/2, while heating up to 1000 °C, the carbides Cr_3C_2 and Cr_7C_3 form (with the content of the latter not exceeding 20 %). That is, it should be noted that carbides can form not only in the reactions of Cr_2O_3 reduction, but also in the places of direct contact between the formed metal Cr and C.

The reduction of oxides $Fe_2O_3{\cdot}Cr_2O_3$ at different ratios of C/Fe and temperatures from 1100 °C to 1250 °C was examined in paper [15]. It was established that with an increase in C/Fe from 0.8 to 1.4, the degree of chromium extraction increased from 9.6 % to 74.3 %. A temperature increase to 1250 °C led to an increase in the formation of carbides. At C/Fe below 0.8, the authors observed a significant decrease in the degree of chromium extraction and carbide formation. It turns out that in order to achieve an increase in the degree of Cr extraction during reduction, there should be a certain excess of C relative to O in the composition of the charge in line with the stoichiometry.

It follows from the considered sources of information that there are significant results in the studies of the processes of Cr reduction on example of separately taken Cr oxides, or a complex of Cr and Fe oxides. However, still insufficiently investigated is the mechanism of the course of reducing and carbide forming processes during carbon thermal treatment of oxide chromium-containing ore raw materials. In contrast to pure oxides of Cr and Fe, the ore raw materials contain related oxide impurities of Mg, Al, Si, Ca, and others. They can significantly affect the flow of reduction processes, as well as phase composition and microstructure of the received products. Research in this direction can result in reducing the losses of Cr during thermal treatment of oxide chromium-containing ore raw materials and subsequent utilization of the obtained alloying additive.

Based on the analysis of scientific literature, it is expedient to undertake a comprehensive study into phase composition and microstructure of the original oxide chromium-containing ore raw materials and the products of carbon-thermal reduction. In addition, the use of raster electron microscopy with an X-ray micro analysis would greatly expand the understanding of the nature of separate phases and inclusions in the studied materials.

3. Research goal and objectives

The goal of present work was to study the properties of oxide chromium-containing ore raw materials and products of carbon-thermal reduction, obtained under experimental-industrial conditions. This is necessary to identify the parameters that reduce the losses of Cr at processing the ore materials and when using briquetted metallized chromium-containing alloying additives in the steelmaking industry. A research in this direction is also required for the further development of understanding the mechanism of the course of reducing and carbide-forming processes during metallization of oxide chromium-containing materials.

To accomplish the set goal, the following tasks had to be solved:

– to determine the features of microstructure of oxide, and the chemical composition of chromium-containing, ore raw materials as the starting substance for carbon-thermal treatment;

– to examine phase composition and microstructure of the briquetted chromium-containing alloying additive after carbon-thermal treatment, received by experimental industrial technique, on the effect on the reduction of losses in the target element during alloying.

4. Materials and methods for the study of properties of oxide chromium-containing ore raw materials and the products carbon-thermal reduction

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

The main component of the charge is industrial finely dispersed chromic ore. We used as a carbon reducing agent the ultra-dispersed lamp black with a carbon content close to 100 % by weight. Obtaining the briquettes was implemented under industrial conditions at a pressure of 12–16 MPa that enabled the assigned density in the range from 2.64 to 3.55 g/cm^3. We used as a binder the carbon organic compounds of petroleum distillation heavy fractions. Parameters of the briquettes: diameter – 0.245 m; height – 0.250–0,350 m; weight – 18–25 kg. The proposed technical solution implied the O/C ratio in the charge at 1.05–1.15, and a thermal treatment mode at 1273 K for 4 hours of isothermal aging, which provides for the obtaining of a new type of alloying material in line with TU 322-297-04-96.

X-ray structural phase analysis of the samples was performed on the diffractometer “DRON-6” (Russia).

Images of the microstructure of samples were obtained from the raster electron microscope “REM-106E” (Ukraine). The microscope is equipped with a system of X-ray microanalysis that determines chemical composition of separate plots on the samples’ surface.

4. 2. Procedure for determining the indicators of samples’ properties

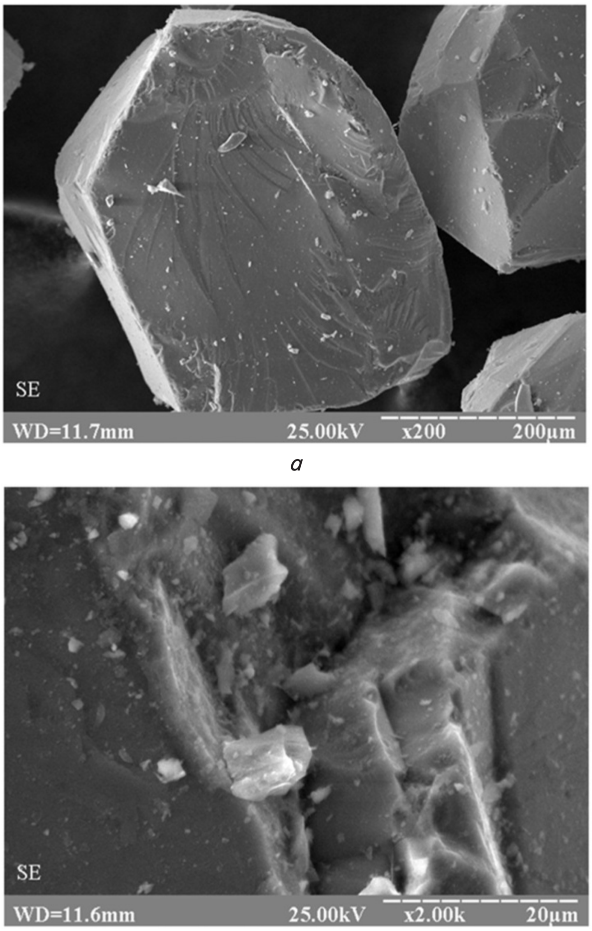
Phase composition of the examined samples was determined using the method of X-ray structural analysis that involved the Cu monochromatic radiation K_α (λ=1.54051 Å). The measurements were performed at a voltage on the tube U=40 kV and anode current I=20 mA. Composition of the phases was determined using the programming package PDWin 2.0 (Russia).

Study into microstructure of the samples was carried out in at accelerating voltage 10–25 kV and at electronic probe current 52–96 mcA. Working distance to the examined surface was 10.5–11.7 mm.

Determining the composition of phases was performed using a reference-free method of calculating the fundamental parameters: computation of correction factors for the reflection of probe electrons, x-ray characteristic absorption and fluorescence. We determined chemical composition of the phases on the areas denoted in the images of the microstructure by the corresponding conditional symbols.

5. Results of studying the indicators of properties of oxide chromium-containing ore raw materials and the products of carbon-thermal reduction

The oxide chromium-containing ore raw material is represented by granules of rounded multifaceted shape with dense monolithic structure. Size of the granules was within 250–600 µm (Fig. 1, *a*). In this case, no bond exists between the granules. Typical quasi-crystalline chippings and cracks on the granules indicate their enhanced fragility (Fig. 1, *b*). The images also exhibited micro particles the size less than 10 µm, which probably broke away from the main particles and attached to the granule surfaces. In addition to Cr and Fe, the oxide chromium-containing ore material revealed the presence of Mg, Al, Si, Ca, and T, which are included in the oxide ore impurities. Chemical composition of the examined area as shown in Fig. 1, *b*, % by weight, is, respectively, Cr – 25,21; Fe – 11.62; Mg – 4.45; Al – 4.20; Si – 0.54; Ca – 0.22; Ti – 0.29; O – 53.47. The corresponding spectrogram of X-ray micro analysis is shown in Fig. 2.



a

b

Fig. 1. Images of the microstructure of oxide chromium-containing ore raw materials at magnification: *a* – ×200, *b* – ×2000

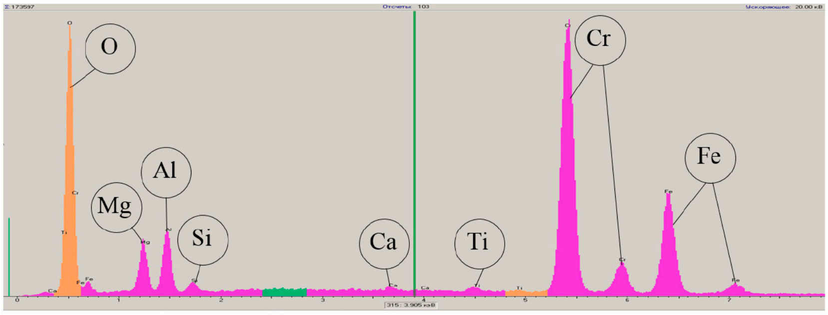
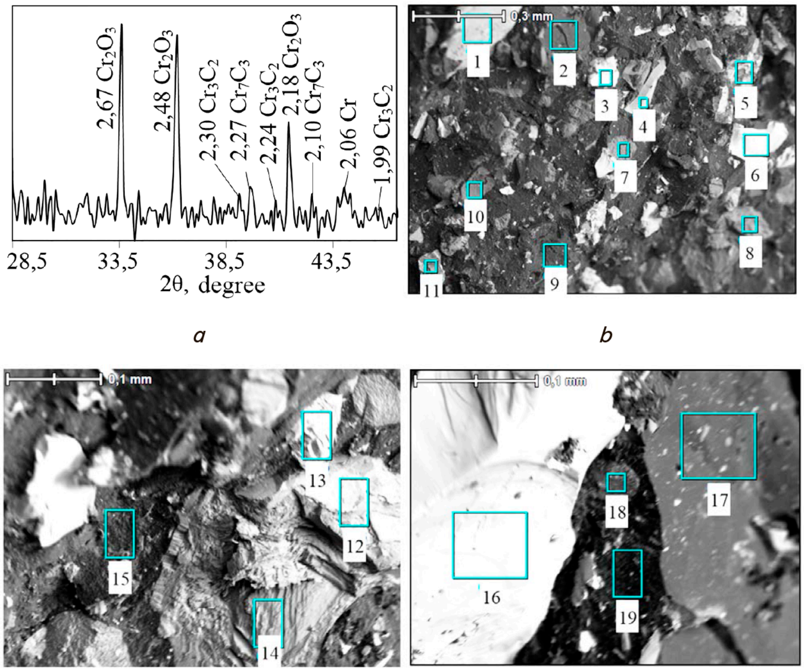


Fig. 2. Spectrogram of X-ray micro analysis of oxide chromium-containing ore raw material in accordance with Fig. 1, *b*



a *b*

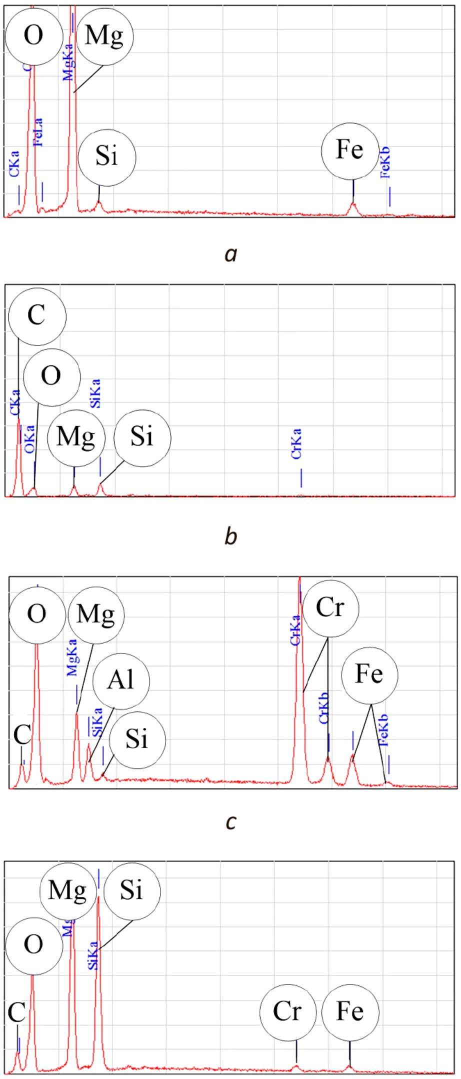
c *d*

Fig. 3. X-ray structural phase studies and microstructure images of the chromium-containing briquette, obtained by the carbon-thermal technique, at different magnification: *a* – a fragment of diffractogram, *b* – ×100, *c* – ×300, *d* – ×400

Results of the phase analysis indicate the presence in the chromium-containing alloying material of Cr_2O_3. Respective diffraction maxima were clearly manifested and had a relatively high intensity (Fig. 3, *a*).

Along with this, the presence of metal Cr is clearly defined. We also found diffraction maxima of Cr_7C_3 and Cr_3C_2. However, they were characterised by a relatively low intensity. The phases of metallic iron, as well as its oxide and carbide compounds, did not explicitly manifest themselves in the diffractogram. This is probably explained by the partial substitution of Cr atoms with Fe atoms in the identified chromium-containing compounds and phases.

The chromium-containing briquette obtained by the carbon-thermal technique, judging by the results of microscopic studies and X-ray micro analysis (Fig. 3, *b*–*d*; Fig. 4; Table 1), had an oxy-carbide structure. The identified particles with a relatively high content of Cr and Fe – 45.48...65.10 % by weight and 13.44...16.13 % by weight (regions 1, 3–6, 11, 13, 16), respectively. The content of the residual O and C was within a range of 7.84...14.35 % by weight and 1.82...8.31 % by weight, respectively. Taking into account the complex of studies conducted, these particles with a high probability consist of oxy-carbides of Fe and Cr.



a

b

c

d

Fig. 4. Spectrograms of certain specified areas according to Fig. 3:
a – 14, *b* – 15, *c* – 16, *d* – 18

We established the presence of particles with a relatively high content of elements that belong to the ore impurities:

Mg, Al, Si, Ca (regions 7, 8, 10, 12, 14, 18). These areas are obviously dominated by the oxide compounds of Mg and Si. At the same time, we identified the areas, which probably represent the unprocessed carbon reducing agent (regions 2, 9, 15, 17, 19), with a carbon content of 60.39...66.68 % by weight.

Table 1

Results of X-ray micro analysis of the sample of a chromium-containing briquette, obtained by the carbon-thermal technique, according to Fig. 3

Region	Content of elements, % by weight									
	C	O	Mg	Al	Si	S	Ca	Cr	Fe	Total
1	4.35	10.11	6.91	3.05	0.72	0.00	0.00	59.89	14.97	100.00
2	66.44	17.38	5.35	0.51	4.86	1.02	0.00	4.44	0.00	100.00
3	1.82	14.35	9.60	4.65	0.80	0.00	0.00	54.62	14.16	100.00
4	8.31	12.86	7.53	3.15	1.38	0.00	0.00	53.33	13.44	100.00
5	3.34	13.70	12.75	1.80	7.35	0.00	0.00	45.48	15.58	100.00
6	1.90	12.36	7.52	4.32	0.00	0.00	0.00	59.43	14.47	100.00
7	9.93	37.12	26.39	0.00	26.56	0.00	0.00	0.00	0.00	100.00
8	7.00	36.86	30.34	0.00	20.54	0.00	0.00	0.00	5.26	100.00
9	65.83	10.20	4.85	0.56	7.02	1.39	0.00	5.12	5.03	100.00
10	13.30	33.69	24.79	0.00	24.97	0.00	0.00	0.00	3.25	100.00
11	2.31	9.39	6.76	2.57	0.41	0.00	0.00	63.52	15.04	100.00
12	7.16	39.57	33.51	0.00	14.65	0.00	0.00	0.00	5.11	100.00
13	3.77	7.84	4.63	2.17	0.36	0.00	0.00	65.1	16.13	100.00
14	3.25	36.93	46.61	0.00	1.21	0.00	0.00	0.00	12.00	100.00
15	60.39	16.27	7.78	0.00	9.63	0.00	0.00	5.93	0.00	100.00
16	2.62	9.85	6.42	3.03	0.49	0.00	0.00	62.81	14.78	100.00
17	66.68	14.72	5.23	0.44	4.25	0.96	0.81	5.1	1.81	100.00
18	11.98	24.6	25.27	0.00	27.18	0.00	0.00	4.29	6.68	100.00
19	66.44	9.42	4.86	0.5	10.85	1.57	0.00	4.23	2.13	100.00

6. Discussion of results of examining the indicators of properties of oxide chromium-containing ore raw materials and the products of carbon-thermal reduction

Results of the studies revealed a relatively small particle size of the oxide chromium-containing ore material. This in turn facilitates the processes of carbide formation because, based on the results of articles [13, 14], carbide-forming processes are of diffusion character. We established the presence in the elements chemical composition of oxide ore impurities of Mg, Al, Si, Ca, and Ti. In other words, these impurities may indirectly affect the course of reducing and carbide-forming processes.

Using the lamp black has confirmed effectiveness of this carbon reducing agent, which is consistent with the results of paper [12]. The presence of metal Cr and carbides of Cr_7C_3 and Cr_2C_3 in the phase composition of carbon-thermal treatment products of oxide chromium-containing ore raw materials agrees well with the results of research reported in articles [13–15]. The results of research presented in paper [10] regarding the impossibility of obtaining a carbon-free product, reduced by the carbon-thermal technique, are confirmed.

A transition of part of Cr_2O_3 to metal Cr metal and a larger, relative to stoichiometry, content of C provides increased reducing ability of the obtained alloying additive. Post-reduction of the oxide chromium-containing component will occur directly during introduction of the received material to molten metal as an alloying additive. This is carried out due to the excess of carbon from carbide and oxy-carbide compounds, as well as from the identified areas of the samples with unprocessed carbon reducing agent. The content of S from 0.96 to 1.57 % by weight in some local areas does not lead to exceeding the permissible norms in general for the sample.

Briquetting the charge at a pressing pressure of 12–16 MPa enabled achieving the indicators of required density in the range of 2.64 to 3.55 g/cm^3. Resulting indicators of the alloying additive allowed us to apply various methods for alloying the steel. This was implemented at Nikopol South Pipe Metallurgical Plant (Ukraine) in the workshop of a pipe instrument during smelting of steel 40HL in the furnace DSP-8 with the main magnesite lining. The following techniques of alloying were used:

1) into the batch under the charge;
2) after melting and loading the slag;
3) on the slag (chromium-containing briquettes were immersed under the slag and remained at the interface “slag-metal”. This made it possible to bring down the losses of chromium by reducing the influence of oxygen of the furnace atmosphere);
4) bringing the melt of metal in a ladle to the assigned chemical composition.

One may note as a shortcoming the lack of research into regularities of influence of technological parameters on the required density of the resulting reduced material in wide ranges. Research into this area could provide the most favorable conditions for the dissolution and absorption of an alloying additive in a liquid metal. Undertaking such studies is appropriate in the future.

Obtaining and using a new alloying material has a positive effect on the reduction of ecological stress in industrial regions. This is predetermined by a decrease in resource and energy consumption and harmful emissions into the atmosphere when compared to traditional technologies of alumo- and carbon-silicon thermal melting.

The use of chromium-containing briquettes obtained by the carbon-thermal technique, with a content of Cr at 35.5 % by weight, was implemented when smelting the tool steel 40HL and it did not lead to any technological difficulties [16]. The smelting was performed in the arc furnace DSP-8 with the main lining using the remelting method for non-alloyed waste of pipe production. Oxy-carbide form of the presence of the main elements Cr and Fe with excessive residual carbon content enabled the assimilation of chromium by the molten steel at the level exceeding 95 %. This indicator is higher compared to that when using standard ferrochromium of the brands FH650 and FH800 – 88–92 %. In this case, the through-extraction of Cr from ore by the smelting redistribution of standard ferrochrome is much lower. The degree of chromium extraction in the ferroalloy production of carbon brands of ferrochrome is about 0.85. In the production of chromium-containing briquettes by the carbon-thermal technique, the losses of chromium may occur mainly during machining of the charge components. The degree of Cr transition to briquette is not less than 0.99 [16].

7. Conclusions

1. The original oxide chromium-containing ore raw materials are represented by granules of rounded shape the size

of 250–600 μm. We found quasi-crystalline chippings and cracks on the granules. On the granule surfaces we observed in some places micro particles the size less than 10 μm. Along with Cr (25.21 % by weight) and Fe (11.62 % by weight), the examined material revealed elements of Mg, Al, Si, Ca, and Ti. They probably were included in the composition of oxide ore impurities.

2. Phase composition of the briquetted chromium-containing alloying additive after carbon thermal treatment, received by industrial-experimental technique, consists mainly of Cr_2O_3. We clearly detected the presence of metal Cr. The carbide compounds of chromium revealed Cr_7C_3 and Cr_2C_3. Diffraction maxima of metal Fe and its compounds did not manifest themselves explicitly, indicating the presence of Fe as substitution atoms in the chromium-containing phases and compounds. The microstructure is heterogeneous. We identified phases with relatively high content of Cr and part of the related ore impurities. The investigated areas with a higher content of C may be the remains of the unprocessed carbon reducing agent. The presence of elevated residual carbon, enabled by the estimated O/C ratio of 1.05...1.15, makes it possible to perform the post-reduction of oxide chromium-containing component directly in a liquid bath while alloying. This provides additional protection from secondary oxidation, thus reducing the irreversible losses of the target element.

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