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DEFINING THE FEATURES OF STRUCTURAL AND PHASE TRANSFORMATIONS IN THE RECYCLING OF ANTHROPOGENIC METALLURGICAL WASTE CONTAINING REFRACTORY ELEMENTS

This paper reports a study into the features of the phase composition and microstructure of a master alloy obtained by using the reduction melting of oxide man-made waste. That was necessary to define those technological indicators that provide for an increase in the degree of extraction of alloying elements during the recycling of anthropogenic raw materials and the subsequent use of the alloying material. It has been determined that the phase composition of the alloy at a Si:C ratio in the charge of 0.11 mainly consisted of a solid solution of elements in α-Fe, as well as carbides Fe_3C and Fe_3W_3C. At the Si:C ratios in the charge of 0.28 and 0.52, along with a solid solution of the elements in α-Fe, Fe_8Si_2C, Fe_5Si_3, and FeSiC, $FeSi_2$ manifested themselves, respectively. The microstructure of the alloy demonstrated a clear manifestation of several phases with different content of alloying elements. Changing a Si:C ratio in the charge from 0.11 to 0.28 and 0.52 led to an increase in the residual silicon content (wt %) in the studied areas, from 0.00–0.25 to 0.12–1.79 and 0.20–2.11, respectively. At the same time, the carbon content (wt %) in the examined areas varied from 0.25–2.12 to 0.24–2.52 and 0.45–2.68, respectively. The content of alloying elements in the investigated areas varied within (wt %): W – 0.00–43.06, Mo – 0.00–32.72, V – 0.19–20.72, Cr – 0.69–33.94, Co – 0.00–3.96. Analysis of the study's results reveals that the most acceptable ratio of Si:C in the charge is 0.52. In this case, there is a certain content of residual silicon along with carbon in the form of carbosilicide and silicide compounds. Such indicators of the alloy provide sufficient reducing capacity of the alloy when used. The properties of the alloy make it possible, when smelting steels, to replace part of those standard ferroalloys that do not have strict carbon restrictions

Keywords: dross of alloy steels, oxide anthropogenic waste, reduction melting, X-ray phase studies

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1. Introduction

Extraction of alloying elements during the recycling of anthropogenic waste is one of the important sources of replenishment of the need for alloying additives. In practice, significant amounts of waste are not recycled effectively enough. The importance of the use of secondary raw materials increases in proportion to the depletion of sources of raw materials, which causes an increase in the prices for alloying materials in the world market [1]. Oxide waste from fast-cutting steel production includes a set of alloying elements, the peculiarities of which interaction must be taken into consideration when defining the technological indicators of processing. This causes difficulties in achieving the manufacturability of the production process and the acceptable cost of production, which is an essential indicator for achieving competitiveness in the domestic and world markets. In turn, ensuring competitiveness involves not only assessing internal and external environments. It is also necessary to apply methods for determining ways to strengthen existing and create new competitive advantages [2]. Such competitive advantages include ensuring conditions for efficient processing and return to production of alloyed anthropogenic waste, which is a source of high-cost elements for steel production.

The above renders relevance to those studies in the field of steel making that address the issue of reducing the loss of alloying elements during the recycling of anthropogenic waste. In order to tackle this task, it is necessary to identify the features of structural and phase transformations that occur during the processing of man-made waste.

2. Literature review and problem statement

The composition of dross from the production of non-alloy steel includes FeO, Fe_2O_3, and Fe_3O_4, which was reported by the authors of [3]. In addition, when investigating the dross of fast-cutting steel, the authors of [4] found WO_2 and $W_2C{\cdot}Mo_2C$. That can be explained by a relatively high degree of alloying. The reduction of dross in a carbothermic way led to the formation of phases of solid carbon solution in the lattice of iron, Fe_3C, and C. In the case of alloyed waste containing, among other, Cr and Mo, the authors [5] obtained similar indicators to the detection of Fe_3C. That indicates that the alloying elements are mostly in the form of substitution atoms in the iron-containing phases. The disadvantage to be considered is the lack of results involving silicon, as a deoxidizer, in combination with carbon. The unresolved part of the problem is associated with determining the features of carbide- and silicide formation in the reduction of alloyed waste.

After the reduction melting of alloyed anthropogenic raw materials with the presence of silicon and carbon, a phase of a solid solution of elements in the lattice of iron and Fe_3C was formed, which was shown in [6]. As a difference, the presence of W in the man-made raw materials, as reported in [7], had an impact on the nature of tungsten-containing formations in the microstructure of reducing smelting products, which must have been of carbide or silicide origin. In phase composition, together with a solid solution of elements in α-Fe, as well as Fe_3C, Fe_3W_3C carbides, the iron silicides FeSi and $FeSi_2$ were observed, as well as iron and tungsten intermetallics. In [8], the silicide FeSi was also detected in the Fe-Cr-C-Si system when studying a coating from the alloy 45Fe39Cr6C10Si. The disadvantage is the lack of research on the properties of recovered raw materials with different amounts of silicon and carbon in the charge. Parts of the problem that remain unresolved relate to the identification of the most rational indicators in the recovery of anthropogenic waste in the Fe-W-Mo-V-Cr-Co-O-Si-C system.

The reduction of the complex compounds $FeO{\cdot}V_2O_3$ and $FeO{\cdot}Cr_2O_3$ at 1,373–1,523 K at different C:Fe (atomic ratio) was described in [9]. An increase in the degree of extraction of vanadium was achieved, from 10.0 % to 45.3 %, chromium – from 9.6 % to 74.3 %. More intensive carbide formation was manifested at 1,523 K. The recovery of chromium from ore raw materials at temperatures from 1,389 K to 1,661 K involving Si and SiC is reported in [10]. The reduced products included metal phase particles containing Cr, Fe, and Si. The authors of [11] detected, in the Mo-O-C system, two-stage transformations at the primary transition of MoO_3 to MoO_2, which later passed into Mo and carbides. A similar sequence of transformations was observed in the W-O-C system [12]. However, the difference is that $WO_{2,72}$ was also found when converting WO_3 as an intermediate oxide besides WO_2. The disadvantage to be considered is the lack of results in the reduction of complex oxide compounds that may be included in the composition of anthropogenic waste. This feature of the presence of elements can cause differences in the progress of reduction reactions. Part of the problem that has remained unresolved concerns studies into the structural and phase composition of man-made waste with a complex content of refractory elements that would require X-ray analysis and microscopy.

One can distinguish the results obtained in studying the features of the composition and processes of recovery with the participation of carbon oxide waste of non-alloy steels [3] and with the content of refractory elements [4, 5]. In the materials obtained, there was a phase of the solid solution of elements in the lattice of iron, residual carbon, carbide and oxide components. The participation of silicon in the reduction processes shown by the authors of [6, 7] led to the development of silicide formation. Studies into the relationship of elements in the Fe-Cr-C-Si system were reported in [8, 10]. The course of reduction together with the formation of carbides in the V-Cr-Fe-O-C system is described in [9], while the transformations in the Mo-O-C [11] and W-O-C systems [12] proceeded in two stages with the formation of axillary oxides. However, it remains unresolved to identify the features of the structural-phase composition of the restored anthropogenic raw materials with a set of refractory elements at different carbon and silicon content in the charge. Varying the ratio of these components opens up the possibility of achieving the required properties of the target material. Applying the latest methods of X-ray phase analysis and electron raster microscopy making use of an X-ray microanalysis apparatus would deepen the idea of the nature of elements in the composition of the recovered raw materials. That, in turn, could help define the acceptable technological parameters for processing and reduce the loss of the contained alloying elements in the process of obtaining and using the alloy.

3. The aim and objectives of the study

The purpose of this work was to identify structural and phase transformations in the processing of anthropogenic metallurgical waste from the production of fast-cutting steels with a complex content of refractory elements when reducing it with carbon and silicon to obtain a master alloy. This is necessary to define the appropriate technological parameters that provide for a decrease in the losses of the main elements of the alloy when obtaining and using as an alloying additive.

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

– to investigate the peculiarities of phases in the alloy when changing the ratio of silicon to carbon in the charge;

– to determine the features of the microstructure of the alloy while examining the content of elements in the phase formations when changing the ratio of silicon to carbon in the charge.

4. The study materials and methods

4. 1. The examined materials and equipment used in the experiment

The starting raw material was dross from the production of fast-cutting steels of tungsten-molybdenum-cobalt-containing grades. Carbon ultra dispersed dust formed at carbon graphite production sites was used as a reducer while ensuring a mass ratio of O:C in the charge of 1.42. The silicon content in the charge was changed by adding ferrosilicon of the FS-65 brand while adjusting the mass ratio of Si:C in the charge in the range of 0.11–0.52. The shavings from force grinding in the charge performed the function of improving heat exchange at the beginning of heating. Smelting was carried out at a temperature of 1,873–1,913 K in alundum crucibles in a furnace with indirect heating, equipped with coal lining. Cooling was performed at ambient temperature in the crucibles, which were removed from the furnace along with the alloy.

The phase composition of the alloy was investigated using the diffractometer «DRON-6» (Russia).

We studied the microstructure of the alloy using the electron raster microscope «JSM-IT300» manufactured by JEOL (Japan). The microscope was equipped with the apparatus X-MAX80 for X-ray microanalysis made by Oxford Instruments (UK), which was applied to determine the content of elements in phase formations.

4. 2. Procedure for conducting experiments and determining the properties of samples

To determine the phase composition of the alloy, X-ray phase analysis was used in monochromatic radiation Co-K_{α} employing PDWin 2.0 software (Russia). The anode current on the tube was 20 mA, the voltage was 30 kV.

The accelerator voltage when acquiring images of the microstructure was 15 kV with an electron probe diameter of 4 nm. The percentage of chemical elements was determined using a reference-free method for calculating fundamental parameters.

5. Results of studying the properties of the obtained master alloy

5. 1. Determining the features of the composition of phases in the alloy with a different Si:C ratio in the charge

The phases found in the alloy at a Si:C ratio in the charge of 0.11 are represented by a solid solution of the elements in α-Fe, as well as Fe_3C and Fe_3W_3C carbides. Compounds with silicon did not have a clear manifestation on the diffractogram (Fig. 1, *a*).

At a Si:C ratio in the charge of 0.28, along with a solid solution of the elements in α-Fe, Fe_8Si_2C was found, as well as Fe_5Si_3 with a relatively weak intensity of manifestation. At a Si:C ratio in the charge of 0.52, the presence of solid solution elements in α-Fe, FeSiC, and $FeSi_2$ was observed in the phase composition. The latter demonstrated a relatively weak manifestation. Changing the ratio of Si:C in the charge from 0.28 to 0.52 led to an increase in the manifestation of carbosilicide and silicide compounds relative to the solid solution of the elements in α-Fe. The compounds of the alloying elements demonstrated a weak fragmentary manifestation close to the background level.

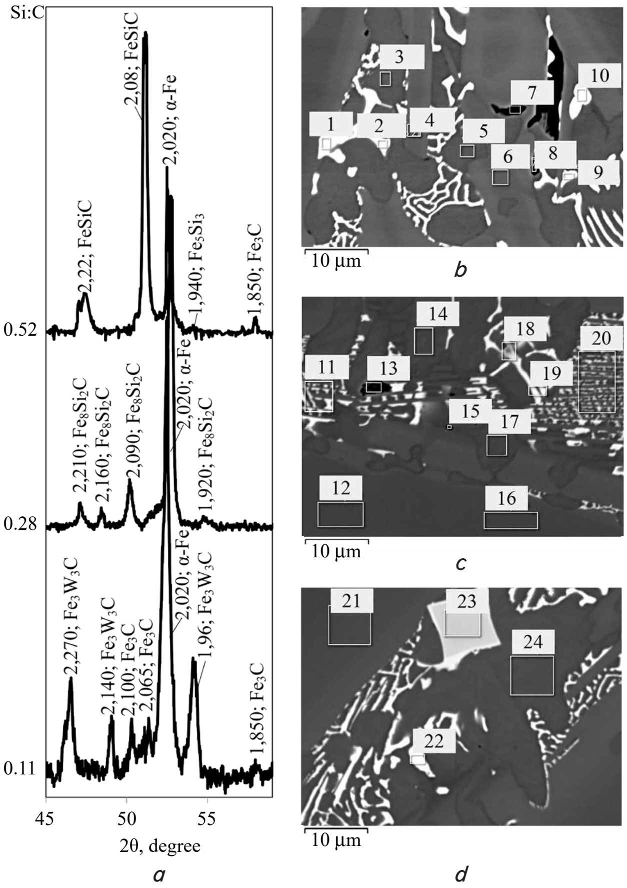

Fig. 1. X-ray phase studies of the alloy at different values of a Si:C ratio in the charge and corresponding images of the microstructure at magnification ×2,500: *a* – diffractograms; *b* – 0.11; *c* – 0.28; *d* – 0.52; 1–22 – regions for determining the composition of elements

5. 2. Studying the features of the microstructure of the master alloy with a different Si:C ratio in the charge

The microstructure of the alloy was characterized by heterogeneity. Several variations of phases with different content of elements were found (Fig. 1–3, Table 1).

A change in the ratio of Si:C in the charge from 0.11 to 0.28 and 0.52 resulted in an increase in the residual silicon content in the studied areas (wt %), from 0.00–0.25 to 0.12–1.79 and 0.20–2.11, respectively. At the same time, the values of residual carbon (wt %) varied from 0.25–2.12 to 0.24–2.52 and 0.45–2.68, respectively.

The content of W (wt %) under such conditions changed values from 0.30–43.06 to 0.00–30.88 and 3.36–40.07, respectively. The Mo content (wt %) – 0.00–32.72 to 0.00–12.28 and 1.28–28.84, respectively. The content of V in the alloy areas within the entire studied interval of Si:C ratios took values in the range of 0.19–20.72 wt %, Cr – 0.69–33.94 wt %, and Co content (wt %) – 0.00–3.96 wt %. Separate residual fragmentary formations with a relatively high oxygen content in the range of 20.47–22.99 wt % at the Si:C ratios in the charge of 0.11 and 0.28. Manganese and titanium in the studied areas manifested themselves fragmentarily at the level of concomitant impurities.

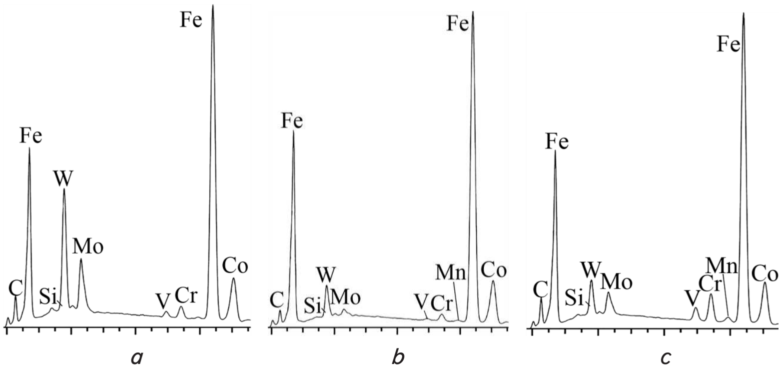

Fig. 2. Spectrograms of X-ray microanalysis of some studied areas of the alloy, according to Fig. 1: *a* — 11, *b* — 17, *c* — 21

Table 1

Results of X-ray microanalysis of alloy areas, corresponding to Fig. 1

No. of entry	Content of elements, wt %											Total
	C	Si	O	Ti	V	Cr	Mn	Fe	Co	Mo	W	
1	2.08	0.22	0.00	1.15	18.37	1.91	0.20	18.79	0.07	17.23	39.98	100
2	2.12	0.25	0.00	0.99	12.90	1.41	0.09	25.02	0.58	17.26	39.38	100
3	0.33	0.00	0.00	0.00	0.19	0.89	0.19	89.89	3.40	1.41	3.70	100
4	0.72	0.09	0.00	0.00	1.65	3.70	0.32	82.70	1.88	3.15	5.79	100
5	0.32	0.00	0.00	0.00	0.24	0.91	0.19	90.33	3.57	1.31	3.13	100
6	0.77	0.12	0.00	0.00	1.75	3.61	0.36	82.96	1.77	3.24	5.42	100
7	0.27	0.00	22.99	0.00	16.04	33.03	2.68	24.58	0.11	0.00	0.30	100
8	0.25	0.00	22.44	0.00	14.35	29.92	2.47	28.69	0.34	0.67	0.87	100
9	1.87	0.14	0.00	0.00	1.09	1.13	0.09	32.22	0.67	21.86	40.93	100
10	1.92	0.17	0.00	0.00	0.78	1.04	0.03	20.10	0.18	32.72	43.06	100
11	2.37	1.73	0.00	0.00	0.71	1.38	0.02	70.14	2.35	7.26	14.04	100
12	0.81	0.35	0.00	0.00	2.22	5.40	0.17	83.22	1.55	2.15	4.13	100
13	0.24	0.00	20.47	0.00	16.32	33.91	2.86	25.81	0.00	0.16	0.23	100
14	0.41	0.17	0.00	0.00	0.31	0.96	0.11	90.79	3.01	1.01	3.23	100
15	0.29	0.00	21.72	0.00	16.17	33.94	2.66	25.22	0.00	0.00	0.00	100
16	0.84	0.37	0.00	0.00	2.07	5.50	0.18	82.96	1.64	2.19	4.25	100
17	0.39	0.12	0.00	0.00	0.25	1.01	0.16	92.05	3.02	1.13	1.87	100
18	2.49	1.52	0.00	0.21	5.95	1.31	0.07	60.09	2.07	7.63	18.66	100
19	2.52	1.65	0.00	0.25	10.44	1.57	0.03	39.2	1.18	12.28	30.88	100
20	2.23	1.79	0.00	0.00	0.67	1.65	0.13	74.93	2.12	5.79	10.69	100
21	0.86	0.42	0.00	0.00	1.73	4.14	0.29	82.75	1.98	2.65	5.18	100
22	2.25	1.85	0.00	0.00	1.14	0.92	0.16	21.62	0.32	28.84	42.90	100
23	2.68	2.11	0.00	1.34	20.72	1.69	0.11	15.72	0.07	15.49	40.07	100
24	0.45	0.20	0.00	0.00	0.20	0.69	0.20	89.66	3.96	1.28	3.36	100

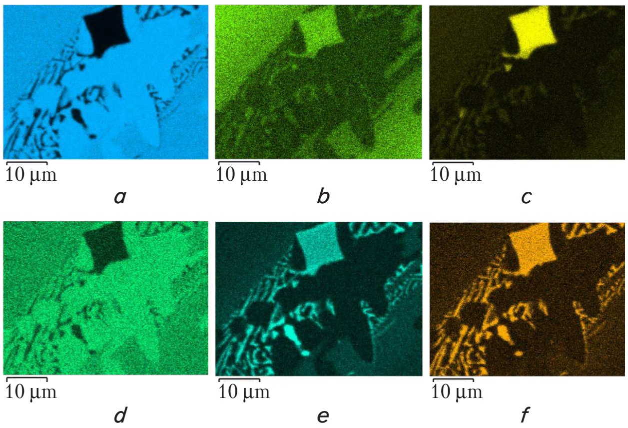

Fig. 3. Distribution of elements in characteristic X-rays according to the results of X-ray microanalysis, according to Fig. 1, *d* (magnification ×2,500). The intensity of color corresponds to the content of elements: *a* – Fe, *b* – Cr, *c* – V, *d* – Co, *e* – Mo, *f* – W

Our study of the distribution of elements in the resulting alloy at a Si:C ratio in the charge of 0.52 (Fig. 3) indicates the combination of areas of the concentration of elements Mo, W, V, Cr.

6. Discussion of results of studying the features of the properties of the obtained master alloy

At different Si:C ratios in the charge, iron-based phases were found in the alloy (Fig. 1). The carbides included Fe_3C, which is in line with the results reported in [3] during the reduction of iron dross. Some of the alloying elements were as substitution atoms in a solid solution in α-Fe, which is consistent with the results reported in [5]. However, the difference in the results of studies [3, 5] is the formation, in addition to Fe_3C, of additional complex Fe_3W_3C carbide at a Si:C ratio in the charge of 0.11. Tungsten compounds were also described in [4, 7]. The presence of Fe_5Si_3 and $FeSi_2$ at the Si:C ratios in the charge of 0.28 and 0.52, respectively, is consistent with the results reported in [6, 8] where iron silicide formation was also observed. As a difference, one can note the formation of carbides, as well as carbosilicides Fe_8Si_2C, FeSiC. This may be due to the higher content of carbide-forming alloying elements. This is also indicated by the results of work [7] where the presence of tungsten had an impact on the nature of phase formations, which was reflected in the phase composition and microstructure. In addition, there is an agreement with the results reported in [12], which indicates the evolution of the formation of tungsten carbides during the reduction of oxide compounds by carbon. Similar processes of carbide formation are observed in the reduction of Mo [11].

During the microstructural studies and X-ray microanalysis of individual phases, a combination of sites with a relatively high content of Mo, W, V, Cr (Fig. 3) was found. That is, unlike the results reported in [11, 12], the possibility of the formation of complex carbide or carbosilicide compounds Mo, W, and other alloying elements does exist. This is in line with the results reported in [9], which noted the formation of carbides in the Fe-Cr-V-O-C system. The combination of the Fe and Co concentration zones (Fig. 3) indicates the latter's dominant quantity as a solid solution in the lattice of α-Fe. Differences in the nature of the presence of elements in the formed particles indicate the presence of several phases of different nature.

Changing the ratio Si:C in the charge from 0.11 to 0.28 and 0.52 resulted in an increase in the residual content of silicon in the corresponding phases in the studied areas of the microstructure (Fig. 1, Table 1). That caused an increase in the manifestation in the phase composition of carbosilicides and silicides in relation to carbides and the solid solution of elements in the lattice of α-Fe. Detecting the fragmentary manifestation of particles with a relatively high oxygen content in the range of 20.47–22.99 wt %. (Fig. 1, Table 1) at the Si:C ratios in the charge of 0.11 and 0.28 indicates the oxide nature of the formations. The manifestation of such formations at a Si:C ratio in the charge is 0.28 less than that at 0.11. One of the reasons may be insufficient number or difficult access of the reducing agent in relation to such sites. It should be emphasized that these particles occur fragmentarily, so, obviously, they do not have a critical effect on the overall target characteristics of the alloy. The post-reduction of such inclusions is possible during the alloying of steel at the expense of the share of residual carbon and silicon in the alloy. With a Si:C ratio in the charge of 0.52, unlike previous cases, no formations with a relatively high oxygen content were found in the structure. At the same time, there is an increase in the manifestation of carbosilicides and silicides, and, accordingly, an increase in the amount of residual silicon, which somewhat limits the flow rate factors of the alloy. On the other hand, an increase in the reducing ability of the alloy is achieved and, as a result, an increase in the degree of extraction of alloying elements. Based on the above, the most favorable Si:C ratio in the charge is 0.52.

Restrictions on the use of alloy can be caused by complex alloying. Complications can occur when some of the components are strictly limited in the target product. Such circumstances may cause the excess of the predefined permissible indicators of the content of the elements in the composition. In order not to face such problems and ensure sufficient cost factors, the proximity of the content of elements in the alloy and the target product should be observed.

As a disadvantage, we can note the absence of the above images of microstructural studies of various magnifications. The presence of such images would add to the demonstration of experiments.

Further advancement of our research could involve expanding the range of anthropogenic waste used in recycling. Difficulties in trying to extend this study relate to an insufficient experimental base.

The resulting alloy is characterized by indicators that make it possible to use it as an alloying additive in the smelting of steel of grades without strict restrictions on carbon to replace part of the ferroalloys that are commonly used. Including ferrotungsten, brand FV70 (GOST 17293-93); ferromolybdenum, brand FMo60 (GOST 4759-91); ferrochrome, brand FX100 (GOST 4757-91); ferrovanadium, brand FVd50U0,4 (GOST 27130-94). Promising, in this case, is the fast-cutting steel of grades R6M5K5, R9M4K8, R12M3K8F2, R10M4K10F3, and others. For smelting, one can use an electric arc furnace with the main lining. Smelting of fast-cutting steel using the alloy is possible under the current technology. In this case, some residual silicon content in the alloy should be taken into consideration. This would affect the amount of adding other silicon-containing components during smelting. The impact of the type of furnace on the potentially expected result may be due to the design features related to the principle of operation of the equipment. For example, the presence of a concentrated heat source over the metal in an electric arc furnace can create local conditions for the burning of alloying elements in such an area. The effect of the chemical composition of the alloy on the results is associated with the content of residual silicon. The relative increase in the silicon content would contribute to increasing the reducing capacity and improving the conditions for the assimilation of alloy elements from the alloy. However, the cost factors of using the alloy are somewhat decreased, due to restrictions on the content of silicon in the steel grades that are smelted.

7. Conclusions

1. It was found that with a Si:C ratio in the charge of 0.11, the alloy demonstrated a solid solution of the elements in α-Fe, as well as Fe_3C and Fe_3W_3C carbides. When the Si:C ratio in the charge changed to 0.28 and 0.52, together with the solid solution of the elements in α-Fe, Fe_8Si_2C, Fe_5Si_3, and FeSiC, $FeSi_2$, respectively, manifested themselves. There was an increase in the manifestation of carbosilicide and silicide compounds.

2. It has been determined that the microstructure of the alloy was characterized by heterogeneity. Several phase variations with different element contents were identified. Changing a Si:C ratio in the charge from 0.11 to 0.28 and 0.52 resulted in an increase in the residual silicon content in the studied areas (wt %), from 0.00–0.25 to 0.12–1.79 and 0.20–2.11, respectively. At the same time, the values of residual carbon (wt %) varied from 0.25–2.12 to 0.24–2.52 and 0.45–2.68, respectively. The content of alloying elements varied within wt %): W – 0.00–43.06, Mo – 0.00–32.72, V – 0.19–20.72, Cr – 0.69–33.94, Co – 0.00–3.96. There were residual fragmentary formations with a relatively high oxygen content in the range of 20.47–22.99 wt %. The investigated phase formations, characterized by a relatively high content of silicon, carbon, and alloying elements, can be carbosilicide compounds.

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