The object of this study is structural and phase transformations during the reduction of high-speed steel scale with carbon to obtain a resource-saving alloying additive. The task addressed is the loss of high-value elements when obtaining and using alloying material from man-made raw materials. Solving the problem is associated with determining technological indicators to enable the reduction of losses of the corresponding elements.

It has been determined that the phase composition of the scale had the manifestation of FeO, Fe_2O_3, Fe_3O_4, $FeWO_4$, MoO_2, FeW_3C, W_2C and Mo_2C. In the reduction products, a solid solution of alloying elements and carbon in α-Fe, residual oxides Fe_2O_3, Fe_3O_4 and carbides FeW_3C, WC, Mo_2C, Cr_7C_3, Cr_3C_2, $Cr_{23}C_6$, VC was found.

The disordered structure of the scale with particles of irregular shape after reduction was characterized by the presence of formations of polyhedral and rounded shapes with different contents of alloying elements. Residual oxygen was within 5.78–7.14% by weight. It was established that achieving a reduction degree of 79% is sufficient since the residual carbon in carbides provides an increased reducing ability during the further reduction of the oxide component in the liquid metal during alloying.

The porous structure causes faster dissolution compared to standard ferroalloys, which reduces the resources consumed by reducing the melting time. No phases with an increased tendency to sublimation were found in the obtained alloying additive. That is, there is no need to create additional conditions that prevent the loss of alloying elements during evaporation with the gas phase, which ensures an increase in the degree of extraction of the corresponding elements.

The properties of the obtained alloying additive allow it to be used in smelting in an electric arc furnace as a replacement for part of standard ferroalloys

Keywords: scale, high-speed steel, oxide technogenic waste, carbon thermal reduction, structural-phase transformations

UDC 669.15'28-198

DOI: 10.15587/1729-4061.2025.337916

ESTABLISHING PATTERNS OF STRUCTURAL-PHASE TRANSFORMATIONS WHEN PROCESSING TECHNOGENIC WASTE OF HIGH-SPEED STEELS BY CARBON THERMAL REDUCTION

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Received 06.06.2025
Received in revised form 18.07.2025
Accepted 19.08.2025
Published 27.08.2025

***How to Cite:** Rebenko, V., Lukianenko, I., Volokh, V., Andreev, A., Poliakov, A., Yamshinskij, M., Zhuravel, D., Ivanchenko, D., Kovalenko, I., Chaplyhin, Y. (2025). Establishing patterns of structural-phase transformations when processing technogenic waste of high-speed steels by carbon thermal reduction. Eastern-European Journal of Enterprise Technologies, 4 (12 (136)), 6–11. https://doi.org/10.15587/1729-4061.2025.337916*

1. Introduction

An alternative source of obtaining refractory alloying materials is the processing of alloyed technogenic waste with its return to production [1]. The latter includes high-speed steel scale. The peculiarity of this type of waste is its fine dispersion and relatively high degree of alloying. This leads to the need to take into account the complex nature of the physicochemical interaction of elements when developing technological conditions for processing. Thus, the relevance of our work is predetermined by the current level of use of alloying elements and the quality of raw materials, which defines the necessary level of competitiveness of metal products in the world market [2].

So, the issue of resource and energy saving with a reduction in losses of alloying elements during the processing of oxide fine-dispersed waste of high-speed steels in metallurgical production is relevant. The priority direction in solving this problem is the development of ideas about structural and phase transformations during carbon-thermal reduction of high-speed steel scale.

2. Literature review and problem statement

According to the studies reported in [3], iron scale consists of Fe_3O_4, Fe_2O_3 and FeO. In this case, carbon reduction was accompanied by the formation of Fe_3C together with a solid solution of carbon in α-Fe. But due to the lack of refractory alloying elements, the problem is the lack of data on the participation of the latter in the reduction process. High-speed steel scale, due to its increased alloying, additionally contains $W_2C{\cdot}Mo_2C$ and WO_2, which is noted in [4]. However, no information is provided on the reduction of technogenic raw materials, which does not allow us to trace the nature of the transformations of the alloying element compounds.

The authors of [5] provided thermodynamic calculations of the carbon thermal treatment of iron oxides for temperatures of 300–1700 K. It was determined that most of the reduction reactions and carbide formation become probable with increasing temperature to 1100 K. It was also noted the possibility of reactions involving CO in chromium carbidization reactions with the formation of Cr_3C_2, Cr_7C_3 and $Cr_{23}C_6$. But there is no possibility to follow the likelihood of reactions with other refractory elements.

The authors of [6] reported research on the carbon thermal treatment of tungsten high-speed steel scale. At different stages of reduction, the transformation of oxide compounds FeO, Fe_3O_4 and $FeWO_4$ into carbides Fe_3C, WC, W_2C, FeW_3C, Fe_3W_3C, Fe_6W_6C, VC, V_2C, Cr_3C_2, Cr_7C_3, $Cr_{23}C_6$ and a solid solution of alloying elements and carbon in α-Fe was observed. At the same time, the $FeWO_4$ phase manifested itself only at the initial stages of reduction. The FeW_3C and Fe_3W_3C carbides manifested themselves with the evolution of reduction process. The W content in the phase formations of the samples ranged from 1.15 to 27.37 wt%. However, due to the absence of molybdenum content, it is not possible to trace the participation of the latter compounds in reduction reactions.

The authors of work [7] studied the carbon thermal treatment of the scale of high-speed steel with a relatively high content of tungsten and vanadium. With the development of reduction processes, the manifestation of a solid solution of alloying elements and carbon in the α-Fe lattice increased. At the same time, the intensity of the diffraction maxima of $FeWO_4$ and Fe_3O_4 decreased. The presence of Fe_3C, FeW_3C, WC, VC, V_2C and Cr_3C_2 was observed in the reduction products. During the reduction process, the manifestation of FeW_3C increased relative to Fe_3C and WC. The W content in the studied areas of the samples varied within 2.36–42.36 wt.%, while the Mo content did not exceed 2.27 wt.%. However, some questions remained unresolved regarding the nature of the presence of conductive elements in phase formations in the reduced complex-alloyed material from the production of high-speed steels with a relatively high content of molybdenum. Part of the unsolved problem concerns the identification of the most acceptable parameters for the reduction of oxide alloyed raw materials in the Fe-W-Mo-V-Cr-O-C system.

The authors of [8] described a thermodynamic analysis of reactions in the Cr-O-C system in the range of 1100–2000 K. When the temperature increased to 1200 K, the formation of Cr_3C_2 was determined, and when the value reached 1300 K, Cr_7C_3. At 1350 K, the formation of Cr was determined in the system. However, it is not possible to trace the influence on the equilibrium of reactions of other refractory elements present in technogenic waste from the production of high-speed steels.

The authors of [9] performed thermodynamic calculations of reactions in the V-O-C system in the range of 300 2000 K. It was determined that the probability of formation of VC carbide was observed already at 900 K and increased with increasing temperature. As a disadvantage, a wider range of alloying elements in the composition of technogenic oxide waste from high-speed steels can be noted, which could lead to the formation of more complex compounds. It is not possible to trace processes of this nature. The unresolved part of the problem concerns the expansion of the scope of ideas about the nature of the presence of elements in the reduction products using scanning electron microscopy and X-ray phase analysis.

In [10], thermodynamic calculations of carbon-thermal reactions involving tungsten and molybdenum oxides are reported. It was determined that the probability of reduction and formation of WC and Mo_2C carbides is observed with reaching 800–1050 K and increases with further increase in temperature. But it is not possible to follow the features of the reduction of raw materials complexly alloyed with refractory elements. As a disadvantage, it can be noted that in oxide waste from the production of high-speed steels, the form of the presence of molybdenum- and tungsten-containing compounds can be more complex, unlike the case with pure oxides. The unresolved part of the problem concerns the determination of technological indicators of the reduction of complexly alloyed technogenic raw materials with the production of products that do not contain phases prone to sublimation. This would eliminate the need to create special conditions to prevent loss of alloying elements by evaporation with the gas phase.

Worth noting are the results of determining the composition of scale from the production of carbon steels [3] and scale containing alloying refractory elements [4], as well as the features of carbon-thermal reduction. After reduction, a solid solution of carbon in the iron lattice, residual oxides and carbides were found. The products of reduction of alloyed scale had a similar composition [1]. A more intense manifestation of iron carbides and alloying elements was determined during the reduction of highly alloyed tungsten-containing [6] and tungsten-molybdenum-containing [7] scale of high-speed steel. Analysis of thermodynamic calculations of reactions in the Mo-O-C, W-O-C systems [10] reveals the probability of the occurrence of reduction reactions and carbide formation. Carbide formation is also observed during carbon-thermal reduction in the V-O-C [9] and Cr-O-C systems [5, 8].

Our review of the literature [1–10] reveals the expediency of conducting research into establishing the features of structural-phase transformations occurring during carbon-thermal reduction of scale of high-speed steel alloyed with tungsten, molybdenum, chromium, vanadium. Achieving the required characteristics of the target product can be ensured by a certain degree of reduction. Reducing the losses of elements present in the material with a gas phase could be enabled by defining technological indicators that would make it possible to obtain a material without components with an increased tendency to sublimation.

3. The aim and objectives of the study

The purpose of our work is to define the structural and phase composition of the scale of tungsten-molybdenum-containing high-speed steel and carbon-thermal treatment products with the identification of transformations in the material during reduction. This is necessary to determine the indicators that reduce the loss of tungsten, molybdenum, and other elements with sublimation during scale processing and the use of a metallized alloying additive in steel smelting.

To achieve this aim, the following objectives were accomplished:

– to define the features of phase composition of the scale of tungsten-molybdenum-containing high-speed steel and carbon-thermal reduction products to determine the processing parameters that exclude the appearance of phases with a high tendency to sublimation;

– to establish the features of microstructure of the scale of tungsten-molybdenum-containing high-speed steel and carbon-thermal reduction products to identify the nature of the presence of elements in the microstructural components in relation to specifying the characteristics of phase formations.

4. The study materials and methods

4. 1. The object and hypothesis of the study

The object of our study is structural-phase transformations during the reduction of high-speed steel scale with carbon to obtain a resource-saving alloying additive.

The hypothesis of the study assumes the possibility of obtaining a reduced product with the presence of carbides without the presence of phases with an increased tendency to sublimation.

The assumption adopted in the study is the possibility to form complex compounds of iron and alloying elements.

The simplification accepted in the work concerns the issue of the possible interaction of the formed various carbide compounds with each other.

4. 2. Materials and equipment used in the experiment

The starting material is scale of high-speed steel of the P6M5 brand. The reducing agent is ultrafine carbon-graphite dust. The addition of the reducing agent ensured the ratio of xenon to carbon in the charge at the level of 1.38. The heat treatment was carried out during isothermal holding at a temperature of 1443 K with the achievement of a reduction degree of 79%. The protective environment is an argon atmosphere.

X-ray phase analysis was carried out using a “DRON-6” diffractometer.

Images of the microstructure together with the content of elements in the studied areas of the sample surface were acquired using a scanning electron microscope “JSM 6360LA” (Japan), which is equipped with an X-ray microanalysis system “JED 2200”, manufactured by JEOL (Japan).

4. 3. Methodology of experiments and determination of sample properties

To determine the phase composition of materials, the method of X-ray phase analysis was used with monochromatic Co Kα and Cu Kα radiation when studying scale and reduction products, respectively. The voltage on the tube was 30 kV, the anode current was 20 mA. The nature of the phases when deciphering the diffractograms was determined using the PDWin 2.0 software.

The image of the microstructure was obtained at an accelerating voltage of 15 kV with an electron probe diameter of 4 nm. The determination of the composition of elements was performed using a reference-free method for calculating fundamental parameters.

5. Results of investigating the properties of high-speed steel scale and reduction products

5. 1. Determining the composition features of the formed phases of high-speed steel scale and reduction products

In the diffractogram of the scale study, FeO was characterized by the greatest intensity (Fig. 1). Fe_2O_3 and Fe_3O_4 had somewhat lower intensity. $FeWO_4$, MoO_2, FeW_3C, W_2C and Mo_2C were also present.

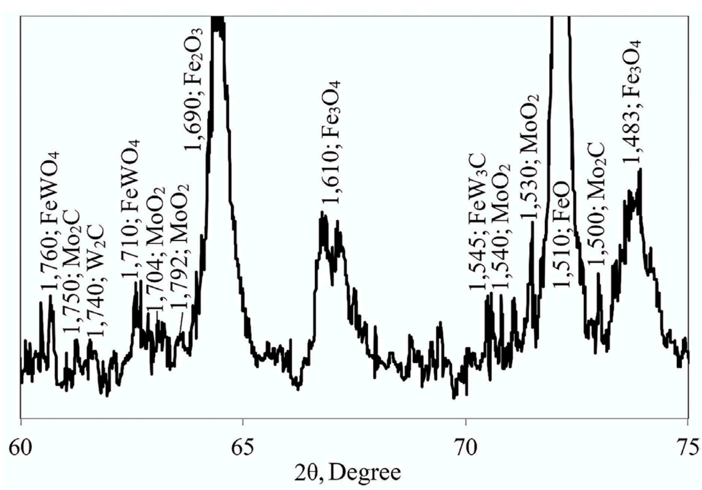


Fig. 1. X-ray diffraction studies of scale

In the phase composition of scale reduction products, the most intense manifestation was the solid solution of alloying elements and carbon in α-Fe (Fig. 2).

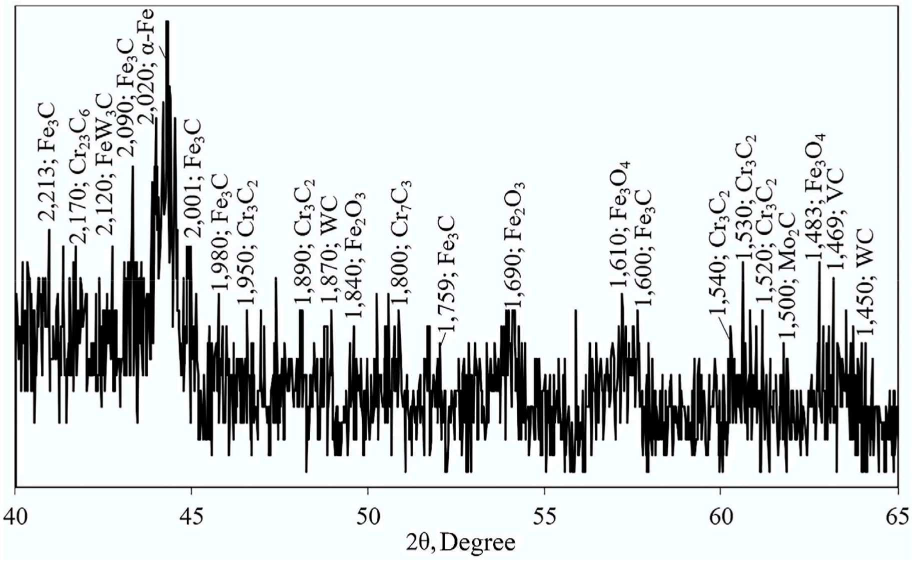


Fig. 2. X-ray phase studies of scale reduction products

Fe_2O_3 and Fe_3O_4 were relatively weaker. The presence of Fe_3C, FeW_3C, WC, Mo_2C, Cr_7C_3, Cr_3C_2, $Cr_{23}C_6$ and VC carbides was detected.

5. 2. Determining the microstructure of high-speed steel scale and reduction products

It was determined that the microstructure of the scale is disordered and represented by particles of irregular shape (Fig. 3, *a*). At the same time, differences were observed in the content of elements in the studied areas (Table 1, Fig. 4). The W content was within 1.76–6.80% wt. The presence of Mo, Cr, and V (% wt.) reached 8.85, 4.07, and 2.36, respectively. The oxygen content was within 26.52–33.71% wt.

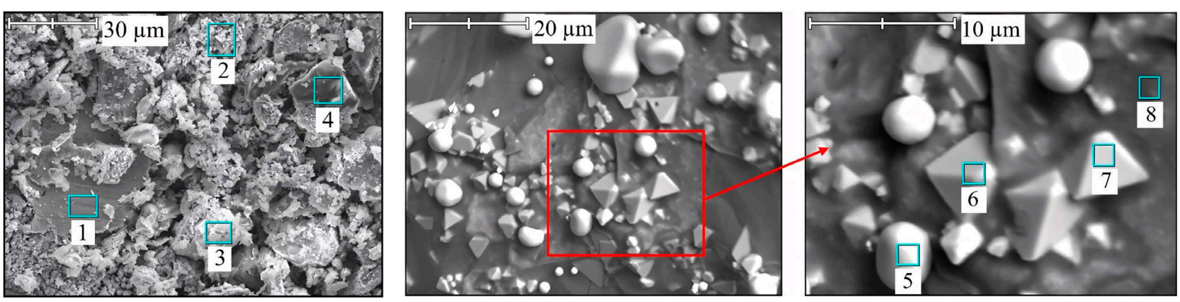
It was found that the microstructure of the reduction products is porous, heterogeneous, and has several types of phase formations. Polyhedral and rounded particles of different chemical composition were detected (Fig. 2, 3, Table 1).

The resulting reduction products had a residual oxygen content in the studied areas within 5.78–7.14 wt%. At the same time, the content of W and Mo was within 1.54–21.75 wt.% and 0.00–11.74 wt.%, respectively. The content of Cr and V was 0.00–9.39 wt.% and 0.00–8.05 wt.%, respectively.

Table 1

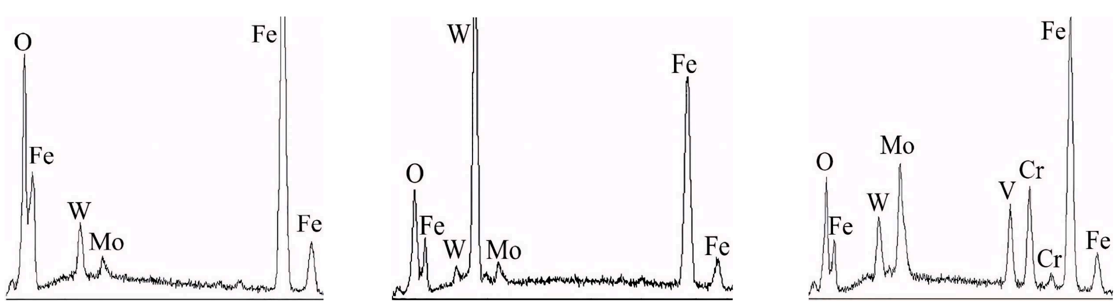
Results of X-ray microanalysis of scale and reduction products according to Fig. 3

No. of order	Element content, % wt.						Total
	O	V	Cr	Fe	Mo	W	
1	33.71	0.00	0.00	64.53	0.00	1.76	100.00
2	28.84	2.36	4.07	52.70	8.85	3.18	100.00
3	26.52	0.00	0.00	63.62	3.06	6.80	100.00
4	31.45	0.00	0.00	58.71	4.69	5.15	100.00
5	5.78	0.00	0.00	69.30	3.17	21.75	100.00
6	6.15	6.13	8.20	63.65	9.58	6.29	100.00
7	6.29	8.05	9.39	56.97	11.74	7.56	100.00
8	7.14	0.00	0.00	91.32	0.00	1.54	100.00



a *b* *c*

Fig. 3. Microstructure images: *a* — scale (magnification ×1000); *b* — reduction products (magnification ×2000); *c* — reduction products (magnification ×5000)



a *b* *c*

Fig. 4. Spectrograms of X-ray microanalysis of certain studied areas of the samples according to Fig. 3: *a* — 3; *b* — 5; *c* — 7

6. Discussion of results based on investigating the properties of high-speed steel scale and reduction products

Our studies indicate that the main phases of the studied scale were FeO, Fe_2O_3 and Fe_3O_4 oxides (Fig. 1), which is consistent with the results reported in [3]. The presence of the above-mentioned oxides as the main phases of high-speed steel scale also agrees with the results from [4]. A similar feature is also the presence of individual compounds of alloying elements. But as a difference, the presence of $FeWO_4$, MoO_2, FeW_3C, W_2C, Mo_2C in the composition of the studied scale should be noted. After carbonization, the phase composition of the reduced raw material was dominated by a solid solution of alloying elements and carbon in α-Fe (Fig. 2). Fe_3C was also detected. This is consistent with the results of thermodynamic calculations of reactions regarding the carbonization of iron oxides, given in [5]. Part of the unreduced component in the form of Fe_2O_3 and Fe_3O_4 oxides remained from the original scale. In contrast to the known results reported in [7], it is possible to note the presence in the phase composition of such carbides as Cr_7C_3, $Cr_{23}C_6$ and Mo_2C. The detection of the latter is also a difference from the results in [6]. At the same time, in contrast to the above-mentioned papers, based on the results of our studies, Fe_3W_3C, Fe_6W_6C, and V_2C were not detected (Fig. 2). Some of the carbides in the reduced raw material could have passed from the original scale. The presence of carbides of alloying elements in the reduced material is consistent with the results of thermodynamic calculations of reactions for the W-O-C [10], Mo-O-C [10], Cr-O-C [5, 8], and V-O-C [9] systems.

Based on the results of microstructural studies of the scale, areas with an increased content of alloying elements were identified (Fig. 3, areas 2–4). They may probably contain oxide or carbide compounds of the corresponding elements, which is consistent with the analysis of thermodynamic calculations of the reactions and the conducted X-ray phase studies (Fig. 1). The relatively high oxygen content additionally indicates the predominance of the oxide component. At the same time, it is not excluded that some of the alloying elements may be present as substitution atoms in iron oxides. From the results of investigating the microstructure of the scale reduction products, some areas with a relatively high content of alloying elements can also be seen (Fig. 3, areas 5–7). The relatively high tungsten content of area 5 (21.75 wt.%) may determine the presence of formed tungsten-containing carbides. This is consistent with the results of the analysis of thermodynamic calculations of reactions, X-ray phase analysis (Fig. 2), as well as with the results reported in [10], which shows the processes of carbide formation during the reduction of tungsten oxides. The detected content of tungsten in the studied areas is relatively lower than the individual indicators in work [6], which is due to the correspondingly different content of the latter in the starting material. In area 5 (Fig. 3), the presence of molybdenum in the amount of 3.17 wt% was also detected. At the same time, a relatively high content of molybdenum (9.58–11.74 wt%) and tungsten (6.29–7.56 wt%) was detected in areas 6, 7. At the same time, molybdenum, like tungsten, is prone to carbide formation reactions during carbonization. The obtained data are consistent with the thermodynamic calculations of the reactions given in [10] and lead to the detection of tungsten-containing carbides WC, FeW_3C, and molybdenum carbide Mo_2C (Fig. 2).

In turn, the molybdenum content in the studied areas is relatively higher than that found in [7], which is due to different brands of the initial scale. Some of the detected formations (Fig. 3, areas 6, 7) were characterized by a relatively high content of chromium (8.20–9.39 wt.%) and vanadium (6.13–8.05 wt.%). Such particles probably contain a complex of chromium and vanadium carbides, which is consistent with thermodynamic calculations of reactions in the Cr-O-C [5, 8] and V-O-C [9] systems. As a disadvantage, a smaller number of components in the studied reaction system can be noted. But as a difference, it can be noted that some of the chromium atoms can be present as substitution atoms in the lattice of iron carbides and α-Fe solid solution. It turns out, taking into account X-ray phase studies, residual carbon in the predominant amount can be in the composition of carbides.

Our set of studies on the phase composition and X-ray microanalysis of the content of metal elements and oxygen in the scale and reduction products is consistent with the considered thermodynamic calculations. The determined possibility of the reduction reactions and carbide formation with the participation of iron and alloying elements corresponds to the identified phases and also determines the relative decrease in the detected values of residual oxygen in the material after heat treatment.

Achieving a reduction degree of 79% ensured a relatively high manifestation of a solid solution of alloying elements and carbon in α-Fe compared to oxides. At the same time, there was a clear manifestation of iron carbides and alloying elements. The residual oxygen in the studied areas of the reduction products was at the level of 5.78–7.14 wt %. According to the results of our studies, acceptable indicators were provided for the subsequent use of the reduction products as an alloying additive. At the same time, due to the residual carbon in the form of carbides, which intensively appeared during X-ray phase studies, a relatively high reducing capacity and level of extraction of alloying elements are achieved. At the same time, the additional reduction of residual oxides occurs in the liquid metal during the alloying process. The porous microstructure contributes to the relatively rapid dissolution of the obtained alloying material compared to standard ferroalloys. This reduces the total melting time, and as a result, reduces the resources consumed.

Some limitations that should be taken into account when trying to apply the results of our research in practice may be associated with the complex alloying of the products of carbonization of high-speed steel scale. Such circumstances apply to the case in which there is a strict limitation of some of the components of the obtained alloying material in relation to the content in the target product. In order to prevent such problems, taking into account the above limitations, and to enable relatively high utilization rates in practice, it is necessary to adhere to a close content of alloying elements in the obtained additive in relation to the target product. The limitations of our results in the field of carbonization of oxide alloyed technogenic raw materials can be taken into account in further research. In the future, in relation to the specified direction, it is possible to perform calculations and analysis of the thermodynamic equilibrium of the set of carbonization reduction reactions and a wider range of alloying elements.

As a disadvantage, one should note the lack of images regarding the microstructure of the initial scale with different degrees of magnification. This would increase the representation of our results.

This direction can be advanced when using for processing oxide technogenic raw materials from the production of other classes of steels. Difficulties in attempts to develop this research were caused by the lack of a sufficient amount of experimental data.

The scope of application of the resulting alloying material includes special metallurgy for the smelting of alloyed steels. The conditions of use correspond to the possibility of using the alloying material for the smelting of steel grades, the composition of which does not have significant restrictions on carbon, to replace part of standard ferroalloys. Taking into account the above, high-speed steels of the grades R6M5, R6M5F3, R6M3 and others, which are smelted in an electric arc furnace, are promising in this case. The addition of the alloying material obtained is possible during filling together with the metal charge. The expected consumption rates of the alloying material in this case can be at the level of 100–200 kg/t of steel, depending on the grade of steel being smelted.

Potentially expected effects from the practical application of our research results, taking into account the accumulated experience, can determine the degree of extraction of alloying elements with values above 90%. In the obtained alloying additive, no phases with a relatively high tendency to sublimation were found. That is, there is no need to provide additional conditions that prevent the loss of high-value elements with the gas phase, which leads to an increase in the degree of assimilation of alloying elements.

Our technique for extracting alloying elements from oxide technogenic raw materials in one's own production could significantly reduce the consumption of standard ferroalloys and other alloying materials.

7. Conclusions

1. We have determined that in the phase composition of the scale, FeO oxide had the greatest manifestation. Fe_2O_3 and Fe_3O_4 were less pronounced. $FeWO_4$, MoO_2, FeW_3C, W_2C and Mo_2C had a relatively low intensity. In the reduction products, a relatively high manifestation of a solid solution of alloying elements and carbon in α-Fe was found compared to Fe_2O_3 and Fe_3O_4. The presence of carbides Fe_3C, FeW_3C, WC, Mo_2C, Cr_7C_3, Cr_3C_2, $Cr_{23}C_6$ and VC was also identified.

2. It was found that the microstructure of the scale is disordered and represented by particles of irregular shape with differences in the content of elements in the studied areas. The W content was within 1.76–6.80 wt%. The presence of Mo, Cr, and V (wt%) reached 8.85, 4.07, and 2.36, respectively. The oxygen content in the studied areas of the scale was within 26.52–33.71 wt%. In the reduction products, particles of polyhedral and rounded shape with different contents of alloying elements were found. The residual oxygen content in the studied areas of the samples was within 5.78–7.14 wt%. At the same time, the W content was within 1.54–21.75 wt%, and Mo – 0.00–11.74 wt%. The Cr and V content in the studied areas of the samples had values at the level of 0.00–9.39 wt% and 0.00–8.05 wt% respectively.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study, as well as the results reported in this paper.

Funding

The study was conducted without financial support.

Data availability

All data are available, either in numerical or graphical form, in the main text of the manuscript.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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