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IDENTIFYING FEATURES IN THE STRUCTURAL AND PHASE COMPOSITION OF THE PRODUCTS OF RECYCLING OF THE SCALE OF HIGH-SPEED CUTTING STEEL BY CARBON THERMAL REDUCTION

Viacheslav Borysov
Corresponding author
Head of Laboratory*
E-mail: nauka4013@ukr.net
Oleksii Torubara
Senior Researcher*
Vadym Volokh
PhD, Associate Professor
Department of Mechanization of Production Processes in the Agro-Industrial Complex**
Anatolii Poliakov
PhD, Associate Professor
Department of Machine Repair, Energy Facilities Operation and Labor Protection**
Mykhail Yamshinskij
Doctor of Technical Sciences, Associate Professor***
Ivan Lukianenko
PhD***
Andrey Andreev
Doctor of Pedagogical Sciences, Associate Professor, Head of Department
Department of General and Applied Physics
Zaporizhzhia National University
Zhukovskoho str., 66, Zaporizhzhia, Ukraine, 69600
Tamara Bilko
PhD, Associate Professor
Department of Department of Livestock Production Mechanization
National University of Life and Environmental Sciences of Ukraine
Heroiv Oborony str., 15, Kyiv, Ukraine, 03041
Dmytro Zhuravel
Doctor of Technical Sciences, Professor
Department of Technical Systems and Technology in Livestock
Dmytro Motornyi Tavria State Agrotechnological University
Zhukovskoho str., 66, Zaporizhzhia, Ukraine, 69600
Dmytro Ivanchenko
Assistant***
*Research Laboratory of Applied Materials Science
Academician Yuriy Bugay International Scientific and Technical University
Mahnitohorskyi lane, 3, Kyiv, Ukraine, 02094
**Volodymyr Dahl East Ukrainian National University
Ioanna Pavla II str., 17, Kyiv, Ukraine, 01042
***Department of Foundry Production
National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute"
Peremohy ave., 37, Kyiv, Ukraine, 03056

This paper reports a study into the features of the structural-phase composition of products from the carbon-thermal reduction of scale of high-speed steels that yields an alloying additive. This is necessary to determine the technological parameters that reduce the loss of target elements in the process of obtaining and using resource-saving alloying material. The study indicates that when the degree of scale reduction changed from 28 % to 67 % and 81 %, an increase in the manifestation of a solid solution of carbon and alloying elements in the α-Fe lattice was observed. At the same time, the intensity of the diffraction maxima of FeO and Fe_3O_4 decreased. In the reduced products, the presence of Fe_3C, FeW_3C, Fe_3W_3C, and WC was traced. With an increase in the degree of scale reduction from 28 % to 67 %, the disordered (of "loose" appearance) microstructure was replaced with the formed particles of round and multifaceted shape with different content of alloying elements. At the reduction stage of 81 %, the microstructure had a finely fibrous structure. Based on the suite of studies, the most acceptable degree of reduction of scale of high-speed steel, followed by the use of the obtained material as an alloying additive, is 81 %. At the same time, ensuring the degree of recovery at the level of 67 % would also suffice. This is due to the fact that residual carbon in the form of carbides provides an increased reducing ability and degree of assimilation of alloying elements with the restoration of the residual oxide component in the liquid metal during doping. Spongy microstructure contributes to faster dissolution, in relation to the corresponding standard ferroalloys. This ensures a reduction in the total smelting time and, as a result, a decrease in the energy consumed

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1. Introduction

One of the significant sources of replenishment of the needs for alloying materials of refractory elements is the processing of man-made waste. At the same time, prices for suitable alloying materials tend to increase [1]. Oxide and fine industrial alloy waste, such as scale of high-speed steels, in practice do not have effective ways of utilization. In such man-made waste, the main alloying elements are contained in the form of complex oxide compounds. Increased doping necessitates taking into account the complex nature of the interaction of alloying elements in determining the technological parameters of processing [2]. These characteristics cause difficulties in the implementation of competitiveness, due to the problems of manufacturability of production and, accordingly, the high cost of final products [3].

Given this, the task to ensure resource saving with a decrease in technological losses of alloying elements at the stages of processing and when using scale of high-speed steels in metallurgical production is relevant. To solve this problem, it is necessary to study the features of the structural-phase composition of carbon reduction products of technogenic oxide raw materials containing refractory elements.

2. Literature review and problem statement

The composition of scale from the production of carbon steel is represented by FeO, Fe_2O_3, and Fe_3O_4, which is shown by the authors of work [4]. Scale of high-speed steel, due to increased alloying, additionally contains $W_2C{\cdot}Mo_2C$ and WO_2, which is reported in [5]. Carbon-thermal reduction of iron scale proceeds through the formation of Fe_3C and the phase of solid carbon solution in the iron lattice [4]. The formation of Fe_3C was also revealed by the authors of work [6] in the study of reducing processes of alloyed man-made raw materials, which contained, among others, Mo and Cr. Alloying elements in predominant quantities were found as substitution atoms in iron-containing phases. As a difference, the additional presence of tungsten in studies of reducing processes, presented by the authors of work [7], influenced the nature of the carbides formed, which had a manifestation in the formation of the microstructure. Separate phase formations were found that were probably carbide in nature and were characterized by a relatively high content of tungsten and carbon with the presence of other elements. But questions remained regarding the nature of the presence of the main elements in the phase formations in the reduced man-made material from the production of high-speed steels. Some unresolved parts of the problem relate to finding the most favorable parameters for the reduction of oxide man-made raw materials in accordance with the Fe-W-Cr-V-Mo-O-C system.

The authors of [8] reported studies of recovery processes in the Mo-O-C system, during which the transformation of MoO_3 to MoO_2, Mo and Mo_2C was observed. At the same time, studies of transformations in the W-O-C system during carbon-thermal reduction, presented by the authors of work [9], indicate the presence of an intermediate stage in the formation of tungsten dioxide. With the development of reducing processes, WO_2 participated in the formation of W and carbides, which is also noted in [10]. As a disadvantage, it can be noted that the nature of the content of molybdenum and tungsten-containing compounds in oxide man-made materials from the production of alloy steels can be characterized by more complex compounds. The parts of the problem that need to be further solved are due to the determination of the parameters of heat treatment of complex oxide material in the production of reduction products that do not contain phases and compounds prone to sublimation. This will avoid the need to create additional conditions during processing, preventing evaporation and loss of high-value elements with the gas phase.

The study of oxide reduction processes in the Fe-Cr-O-C and Fe-V-O-C systems in the temperature range of 1100–1250 °C was carried out by the authors of work [11]. It was shown that when C:Fe changed in the charge from 0.8 to 1.4, there was an increase in the degree of extraction of chromium and vanadium (%) from 9.6 to 74.3, and from 10.0 to 45.3, respectively. Intensification of the processes of carbide formation was observed after heat treatment at 1250 °C [11], the proportion of which is inevitably present in the composition of the reduction products [12]. The course of parallel reduction and formation of carbides Cr_3C_2, Cr_7C_3 and $Cr_{23}C_6$ in the Fe-Cr-O-C system was shown in the results of research by the authors of [13]. As a disadvantage, it can be noted that a larger range of refractory elements in the processed material, inherent in the composition of oxide waste from the production of high-speed steels, could lead to the formation of more complex carbides. There is no way to follow the course of such processes. The unresolved parts of the problem are in the plane of expanding ideas about the nature of the presence of elements in the recovered material using a set of studies involving X-ray phase analysis, raster electron microscopy, X-ray microanalysis.

One should note the results of the study into the composition and patterns of carbon-thermal reduction of scale from the production of carbon steels [4] and alloyed refractory elements [5]. Moreover, the latter had in the composition of the compound alloying elements. As part of the studied materials for the reduction of unalloyed scale, phases such as a solid solution of elements in an iron lattice, iron carbides, and residual oxides were found. Relatively close to the above results were those reported in [6], where the processes of reduction of oxide waste from the production of alloyed steels were investigated. As a difference, according to the results of work [7], the presence of tungsten in the feedstock caused a more pronounced presence of individual structural formations that had signs of a carbide component. During the studies of transformations in the systems Mo-O-C [8] and W-O-C [9], the authors of these works revealed that the reduction takes place through the appearance of lower oxides and the subsequent formation of the metal and carbide components [10]. Parallel carbide formation together with reduction was also observed during recovery processes in the Fe-Cr-O-C, Fe-V-O-C [11], and Cr-O-C systems [13]. There is an inevitable presence of a certain amount of carbides in carbon-thermal reduction products [12]. The analysis indicates the feasibility of performing research aimed at identifying the features of the structural-phase composition of carbon-thermal reduction products of oxide man-made raw materials containing W, Cr, V, Mo. The specified indicators of the target material can be ensured by achieving a certain degree of reduction. Determination of technological indicators that will ensure the production of a product without components with an increased tendency to sublimation will reduce the loss of target elements with a gas phase.

3. The aim and objectives of the study

The purpose of our research was to identify the features of the structural-phase composition of the products of processing scale of high-speed steel carbon-thermal reduction with the production of an alloying additive. This is necessary to find the most acceptable technological parameters for reducing the loss of refractory alloying elements during the production and use of an alloying additive in steelmaking.

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

– to investigate the features of the phase composition of the products of heat treatment of scale of high-speed steel with varying degrees of reduction;

– to determine the features of the microstructure of the products of heat treatment of scale of high-speed steel with varying degrees of reduction with the identification of the content of elements in individual areas of the sample surface.

4. Materials and research methods

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

As a feedstock, scale of high-speed steel of R6M3 grade was used. The reducing agent was ultrafine dust of carbon-graphite production, the addition of which made it possible to achieve an oxygen-to-carbon ratio in the charge at 1.33. Ensuring an appropriate degree of reduction was achieved by isothermal exposure at 1473 K. Argon atmosphere was used as a protective medium.

X-ray phase analysis was performed on the diffractometer "DRON-6".

The image of the microstructure and the content of elements in certain areas of the sample surface were obtained using the raster electron microscope "JSM 6360LA", equipped with an X-ray microanalysis system "JED 2200", manufactured by JEOL (Japan).

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

The phase composition of the materials was determined by X-ray phase analysis. Co Kα monochromatic radiation was used. The anode current on the tube was 20 mA, the voltage on the tube was 30 kV. To determine the nature of the phases, PDWin 2.0 software was used.

Images of the microstructure were obtained at an accelerator voltage of 15 kV. The diameter of the electron probe was 4 nm. Determination of the percentage composition of the main alloying elements and the iron base, as well as residual oxygen in the materials, was carried out using a non-reference method for calculating fundamental parameters.

5. Results of investigating the properties of scale reduction products of quick-cutting steel

5. 1. Determination of the characteristics of the formed phases of the products of reduction of scale of high-speed steel

In the case of a degree of reduction of 28 %, in the phase composition, FeO and Fe_3O_4 were most intensively manifested with a relatively weak manifestation of a solid solution of carbon and alloying elements in the α-Fe lattice (Fig. 1, *a*). Carbides Fe_3C, FeW_3C, and WC were also detected, which indicates a parallel course of the processes of recovery and carbide formation. An increase in the degree of reduction to 67 % led to an increase in the intensity of diffraction maxima of solid carbon solution and alloying elements in the α-Fe lattice with respect to FeO and Fe_3O_4. At the same time, the tendency to reduce the intensity of manifestation of FeO had a more rapid reflection than in the case of Fe_3O_4. Together with Fe_3C, FeW_3C, and WC, Fe_3W_3C carbide was additionally detected. Increasing the degree of reduction to 81 % provided a further increase in the manifestation of solid carbon solution and alloying elements in the α-Fe lattice on the basis of determining the phase composition of scale reduction products.

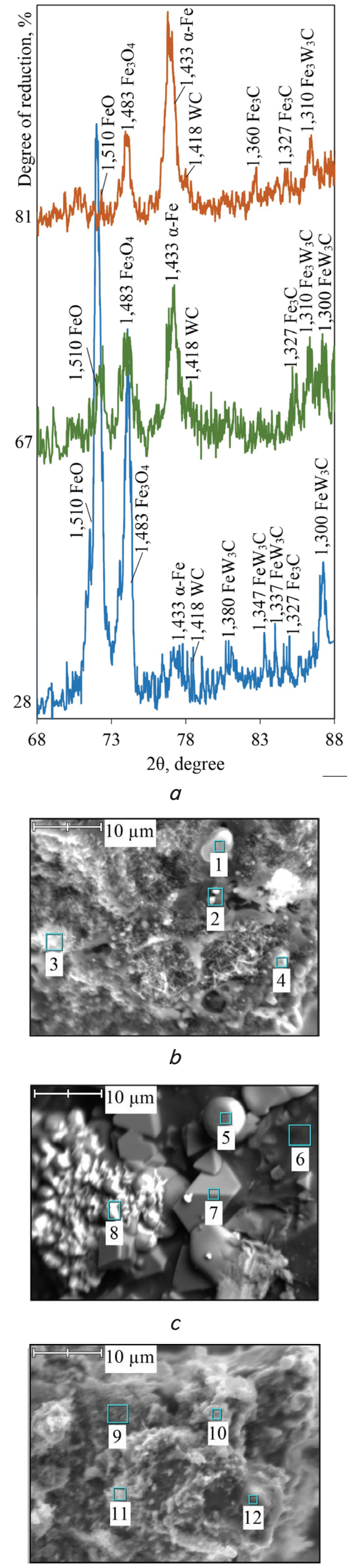

Fig. 1. Areas for determining the phase composition of scale reduction products and corresponding images of the microstructure with varying degrees of reduction at a magnification of ×3000: *a* – diffractograms; *b* – 81 %; *c* – 67 %; *d* – 28 %; 1–12 – areas for determining the composition of elements

The intensity of diffraction maxima of iron oxides at a reduction state of 81 % was characterized by a relatively low level. At the same time, the manifestation of FeO was significantly lower than Fe_3O_4, and had values close to the background level (Fig. 1, *a*). The development of carbide formation processes was expressed in the manifestation on the sites of determining the phase composition of Fe_3C, WC, and Fe_3W_3C.

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

It was revealed that the microstructure is heterogeneous and is characterized by the presence of several variations of phase formations with different content of elements (Fig. 1, 2, Table 1). Upon reaching a degree of recovery of 28 %, the microstructure had an unordered "loose" appearance. The study of sites 9 and 11 (Fig. 1) showed a relatively high content of tungsten (3.74 % wt and 8.76 % wt, respectively), and other alloying elements. This may indicate the presence of complex oxycarbide or carbide compounds with the content of alloying elements. An increase in the degree of recovery to 67 % led to the formation of particles of round and multifaceted shape. Site 8 (Fig. 1) was characterized by a relatively high content of tungsten (9.48 % wt), which may indicate the development of the formation of carbides based on iron and tungsten, such as FeW_3C and Fe_3W_3C. Our study of site 7 showed an increased content of Cr and V (32.35 % by weight and 21.14 % by weight, respectively), which determines the possibility of the presence of complex carbides with the participation of relevant elements. The content of the elements determined at site 5 indicates the formation of iron-containing particles with a relatively low content of alloying elements, which may correspond to the results of the reactions of the formation of Fe_3C. With a degree of recovery of 81 %, the microstructure had a finely fibrous structure. The results of the study of sites 2 and 3 (Fig. 1) indicate the formation of complex phases of iron and alloying. The W content in these areas was at the level of 2.19 % wt and 10.98 % wt, respectively. The presence of Cr was 4.54 % wt and 0.93 % wt respectively, V – 3.21 % wt and 0.71 % wt, respectively. Studies have indicated the possibility of the presence in these areas of both carbides and solid carbon solution and alloying elements in the α-Fe lattice.

Table 1

Results of X-ray microanalysis of scale reduction products based on Fig. 1

No. of entry	The content of elements, wt%						Total
	O	V	Cr	Fe	Mo	W	
1	5.06	0.00	0.00	94.58	0.00	0.36	100.00
2	4.19	3.21	4.54	85.87	0.00	2.19	100.00
3	3.22	0.71	0.93	82.37	1.79	10.98	100.00
4	6.38	0.00	0.74	92.31	0.00	0.57	100.00
5	5.43	0.00	0.00	93.40	0.00	1.17	100.00
6	9.27	0.56	0.73	87.58	0.00	1.86	100.00
7	6.59	21.14	32.35	37.53	0.00	2.39	100.00
8	5.65	2.71	3.14	77.08	1.94	9.48	100.00
9	11.02	6.85	0.48	77.91	0.00	3.74	100.00
10	14.70	0.00	2.01	82.64	0.00	0.65	100.00
11	12.96	1.38	1.66	73.76	1.48	8.76	100.00
12	17.53	0.00	0.00	82.25	0.00	0.22	100.00

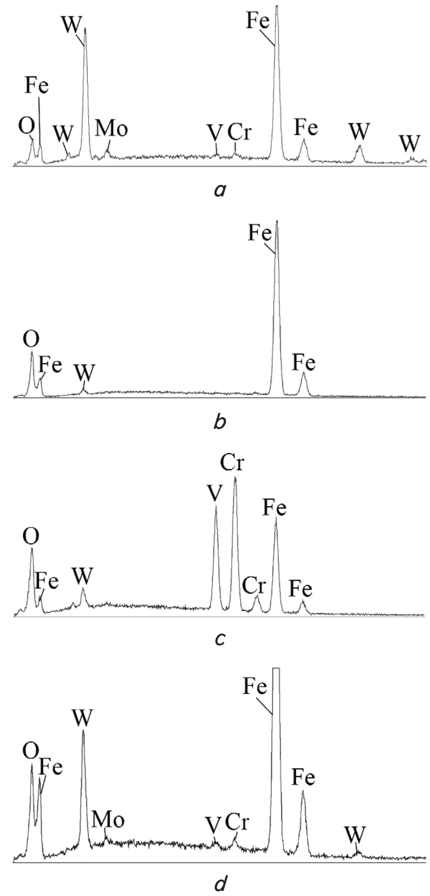

Fig. 2. X-ray microanalysis spectrograms of some studied sample sites, respectively, Fig. 1: *a* – 3, *b* – 5, *c* – 7, *d* – 11

A gradual increase in the degree of recovery from 28 % to 67 % and 81 % led to a relative decrease in the residual oxygen content in the studied areas (wt%) from 11.02–17.53 to 5.43–9.27 and 3.22–6.38, respectively (Fig. 1, Table 1). At the same time, the content of W and Mo (wt%) was in the range of 0.22– 10.98 and 0.00–1.94, respectively. The content of Cr and V in the areas of the samples studied ranged from 0.00–32.35 wt% and 0.00–21.14 wt%, respectively.

6. Discussion of results of investigating the properties of quick-cutting steel scale reduction products

Our studies have shown that the bulk of the phase composition of heat treatment products with varying degrees of reduction was Fe_3O_4, FeO, and a solid solution of carbon and alloying elements in the α-Fe lattice. Iron oxides, as an under-reduced component, came from the initial scale, in which they acted as the main phases, which is consistent with the results of work [4]. As a difference, in relation to the results reported in [6], where only Fe_3C was identified from carbides, in our studies one should note the manifestation in the phase composition of FeW_3C, Fe_3W_3C, and WC. Given the results reported in [5], part of the carbides in the reduction products could be transferred from the composition of the original scale. The relatively high presence in samples of carbide-forming refractory elements, in comparison with studies [7], indicates a significant participation in the processes of interaction of alloying elements with carbon. At the same time, in contrast to work [7], there is a more complex composition of such alloying elements.

Based on the results of the study of microstructure, it is possible to mark areas with a relatively high concentration of alloying elements (Fig. 1, sites 2, 3, 7, 8, 9, 11). A relatively high W content at sites 3, 8, 11 (8.76–10.98 wt%) may indicate the presence of formed oxycarbide or carbide iron-tungsten-containing compounds. That corresponds well to the X-ray phase studies (Fig. 1, *a*), and is also consistent with the results of work [10], which presents the processes of formation of tungsten carbides during the reduction of the corresponding oxides. The presence of W free in the results of [9], as a difference in relation to our studies, is due to the relatively higher content in the feedstock. Together with W, the presence of Mo (1.48 –1.94 wt%) was found in the respective areas, which is consistent with the content of these elements, respectively, of the P6M3 steel grade, in the production of which the initial scale was formed. At the same time, Mo, like W, during a carbon thermal process is prone to the formation of carbide compounds, which is consistent with the results reported in [8] and indicates the possibility of the presence of complex carbide compounds W and Mo.

Some of the formations in the images of the microstructure (Fig. 1, *b–d*, Table 1) were characterized by a relatively high content of Cr (3.14–32.35 wt%, sites 2, 7, 8) and V (2.71–21.14 wt%, sites 2, 7, 8, 9). In such formations, probably, there is a complex of carbides Fe, Cr, and V, which is consistent with the results from [11]. But, as a disadvantage in that work, it is possible to note a smaller set of components in the studied system of reactions without the participation of other refractory elements. The formation of chromium-containing carbide compounds is also consistent with the results reported in [13]. At the same time, as a difference, given the X-ray phase and microstructural studies carried out, part of the Cr atoms can be located as replacement atoms in the lattice of iron-based carbides. That is, the residual carbon in the reduction products is to a greater extent in the form of carbide compounds of iron and refractory elements. This, in turn, is consistent with the results of [12], which indicates the impossibility of obtaining carbon-thermal reduction products without the residual presence of carbides.

Increasing the degree of recovery from 28 % to 67 % and 81 % provided a gradual increase in the intensity of manifestation of solid carbon solution and alloying elements in the α-Fe lattice in combination with carbides in relation to the oxide component. At the same time, residual oxygen in the studied areas of recovery products decreased from 11.02–17.53 wt% to 5.43–9.27 wt% and 3.22–6.38 wt%, respectively. From this position, based on the suite of our studies, the most acceptable degree of reduction of scale of high-speed steel with the subsequent use of the material obtained as an alloying additive is 81 %. At the same time, achieving a degree of recovery at the level of 67 % is also sufficient. This is due to the fact that due to the residual carbon in the form of carbides, which were intensively manifested in the results of research, a relatively high reducing ability and degree of assimilation of the target alloying elements are ensured. The post-reduction of the residual oxide component occurs in the liquid metal during the doping process. The spongy microstructure of the alloying material contributes to relatively rapid dissolution relative to standard ferroalloys. This causes a decrease in the total melting time and, as a result, a decrease in resources spent.

Certain restrictions on the use of carbon-thermal reduction products of scale of high-speed steel may be associated with complex alloying. Difficulties may arise in the case when some of the elements of the resulting alloying material have significant limitations in the target product, which may cause an excess of the established content of elements in the composition. In order to prevent such problems and, at the same time, to ensure relatively high utilization rates, it is necessary to observe the proximity of the composition of the elements of the alloying additive to the target product.

As a disadvantage, we can single out the lack of images of the microstructure, which are obtained with different magnifications. This would provide greater evidence of our studies.

The development of this direction is possible by attracting the use of fine oxide waste in the processing after the production of other steel classes. Difficulties in trying to develop this study consisted in the absence of an experimental base in the required amount.

The indicators of the resulting alloying additive make it possible to smelt grades of alloyed steels, the composition of which does not have significant carbon restrictions, by replacing part of the standard ferroalloys. Taking into account these factors, high-speed steels of grades R6M3, R6M5, R6M5F3 and others, which are smelted in an electric arc furnace, are promising for this. In the resulting alloying additive, no phases and compounds with an increased tendency to sublimation were found. That is, there is no need to create additional conditions that prevent evaporation and loss of high-cost components with the gas phase, which causes an increase in the degree of extraction of alloying elements.

7. Conclusions

1. It was determined that with a gradual change in the degree of reduction of scale of fast-cutting steel from 28 % to 67 % and 81 %, an increase in the manifestation of solid carbon solution and alloying elements in the α-Fe lattice was observed. At the same time, the intensity of the diffraction maxima of FeO and Fe_3O_4 decreased. In the reduction products, the presence of Fe_3C was traced, FeW_3C, Fe_3W_3C, and WC. At the same time, the manifestation of Fe_3W_3C and Fe_3C increased relative to FeW_3C.

2. It was found that with an increase in the degree of scale reduction from 28 % to 67 % to replace the disordered (“loose” appearance) microstructure, the formation of particles of round and multifaceted shape of different content of alloying elements was traced. With a degree of recovery of 81 %, the microstructure had a finely fibrous structure. A gradual increase in the degree of recovery from 28 % to 67 % and 81 % led to a relative decrease in the residual oxygen content in the studied areas (wt%) from 11.02–17.53 to 5.43–9.27 and 3.22–6.38, respectively. At the same time, the content of W and Mo (wt%) was in the range of 0.22–10.98 and 0.00–1.94, respectively. The content of Cr and V in the areas of the samples studied ranged from 0.00–32.35 wt% and 0.00–21.14 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 and the results reported in this paper.

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Data availability

All data are available in the main text of the manuscript.

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