

A STUDY OF ENVIRONMENTALLY SAFE OBTAINING OF MOLYBDENUM-BASED ALLOYING MATERIAL BY SOLID PHASE EXTRACTION

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Визначено кінетичні закономірності вуглецевотермічного відновлення оксидного молибденового концентрату в інтервалі 873–1473 К. Найвищий ступінь відновлення 97 % досягнуто після обробки при 1373 К із переважанням у фазовому складі Мо. Мікроструктура – губчаста розупорядкована. Поліпшення екологічної безпеки досягається заміною вуглецевосиліко- та алюмотермічної плавок феросплавного виробництва Мо на новітні методи порошкової металургії

Ключові слова: молибденовий концентрат, вуглецевотермічне відновлення, металізація, сублімація, фазовий аналіз, мікроструктура, ресурсозбереження

Определены кинетические закономерности углеродотермического восстановления оксидного молибденового концентрата в интервале 873–1473 К. Наибольшая степень восстановления 97 % достигнута после обработки при 1373 К с преобладанием в фазовом составе Мо. Микроструктура – губчатая разупорядоченная. Улучшение экологической безопасности достигается заменой углеродосиліко- и алюмотермической плавок ферросплавного производства Мо на новейшие методы порошковой металлургии

Ключевые слова: молибденовый концентрат, углеродотермическое восстановление, металлизация, сублимація, фазовый аналіз, мікроструктура, ресурсозбереження

1. Introduction

In recent years, the demand for steel alloyed with rare and refractory elements, in particular Mo, has increased.

Prices for the corresponding alloying materials in the world market tend to increase, too. Traditional technologies (carbon monoxide and aluminothermic melting) for obtaining alloying materials based on Mo are characterized by signif-

icant temperatures and processing time. Accordingly, these processes are accompanied by relatively high environmental pollution with gaseous reaction products [1] and solid waste, along with significant resource and energy costs.

Thus, the problem of resource and energy saving with decreasing Mo losses during the processing and using molybdenum-containing ore materials in steelmaking is important. It requires research into the mechanism for recovering molybdenum-containing oxide raw materials. Improvement of environmental safety is possible by replacing the extraction melting with the latest methods of powder metallurgy using solid-state recovery.

2. Literature review and problem statement

The advantages of metallized molybdenum-containing alloying materials in relation to melted ones are mentioned in [2]. The main positive side is the possibility to achieve the dissolution rate and assimilation degree of Mo in the molten metal 2–2.5 times faster. In terms of the methods for the processing of oxide ore and technogenic raw materials in practice, the solid phase extraction of carbon [3] or hydrogen [4] has positive results. The choice of a solid phase extraction method, instead of the retrieving melting process to obtain a molybdenum-containing ligature [5], is environmentally safer as to environmental pollution with waste products and reaction products. The process requires lower temperatures and energy resources, and it is accompanied by significantly less losses of the target element [6, 7].

Research on the kinetics and mechanism of hydrogen restoration of MoO₃ as the main component of a molybdenum oxide concentrate to MoO₂ are presented in [8]. The formation of intermediate oxide Mo₄O₁₁ and parallel reactions of recovery to Mo₄O₁₁ and MoO₂ have been confirmed experimentally.

Hydrogen recovery of MoO₃ at 1323 K is accompanied by the formation of an intermediate MoO₂ product and subsequent production of ultrafine Mo powder of high purity [9]. That is, in papers [8, 9] the stage of reactions with the transition of higher oxides to the lower ones with subsequent restoration to Mo is confirmed. This feature is also presented by the authors of work [10], which also indicates the possible significant impact of the presence of other components in the system of impurities on the nature and characteristics of the recovery. The impurities can also react with the alloying agent and the molybdenum-containing compounds to form joint compounds [10]. The influence of the above-mentioned factors is not excluded in the extraction of molybdenum oxide concentrate, since it contains, together with molybdenum-containing oxide compounds, related ore impurities.

The authors of [11] researched the thermodynamics and kinetics of regeneration in the Mo–O–C and Ca–Mo–O–C systems, which correspond to thermal carbon processing of the molybdenum oxide concentrate. The study determined the formation of MoO₂ as an intermediate product that subsequently became Mo and carbides. The process was conditionally divided into the primary interaction of MoO₃ and C with the course of the C gasification reaction and the subsequent course of MoO₂ recovery with the participation of CO. However, the results of the research in [11] do not give any idea of the effect of the accompanying ore impurities present in the oxide ore concentrate on the recovery process.

This, in turn, can significantly affect the phase composition and the structure of the recovery products.

The products of the reactions obtained in [11] are not subject to sublimation in contrast to MoO₃ [12]. This property of MoO₃ is the cause of significant irreversible losses of Mo when receiving and using molybdenum-based alloying materials. In view of this, when developing the technological parameters for extracting the molybdenum oxide concentrate, the temperatures should be relatively low. However, the temperature should be sufficient for the transition of MoO₃ into the phase and the connection with a relatively low susceptibility to sublimation.

The above-mentioned conditions to decrease Mo losses by sublimation were implemented by the authors of [13] when the MoO₃ powder was restored using a two-stage heat treatment mode. The first stage proceeded at 400–600 °C with the recovery of MoO₃ to MoO₂. The second stage was at 800–1000 °C, providing a recovery of MoO₂ to Mo. The indicated temperature intervals may be tentative in determining the most favorable parameters for the reduction of the molybdenum oxide concentrate.

The proof of the primary propensity to convert MoO₃ to MoO₂, which has a relatively lower propensity to sublimation, is given in [14]. The interaction of MoO₃ powder with CO is observed at relatively low temperatures, starting even at 200 °C [15]. That is, the study confirms the possibility of extracting molybdenum oxides from an oxide concentrate, as well as of carbide formation [16], at relatively low metallization temperatures.

Positive results of using thermal carbon extraction were obtained by utilizing Mo from molybdenum-containing slags formed during the production of copper [17]. The Mo content was about 0.3 % by weight. The efficiency of removing Mo from waste lubricants was presented by the authors in article [18]. The mechanism of thermal carbon extraction was investigated in the temperature range from 1073 K to 1473 K and at 1773 K. A target iron-molybdenum-containing product was obtained with Mo and C contents of about 40 % by weight and 4.3 % by weight, respectively. This further confirms that it is not possible to obtain a carbon-free product by thermal carbon regeneration of oxide molybdenum.

The use of carbon as an alloying agent is technologically simpler and economically more attractive than hydrogen. Yet, in the restoration products, the presence of residual carbon bound in oxycarbide and carbide compounds is possible [19]. This causes some restrictions on the use of the resulting material without further processing for alloying carbon-based steels and alloys. However, the possibility of using a successful plasma processing experience eliminates this disadvantage [20, 21]. In article [20], the authors presented the possibility of obtaining relatively pure Mo powder from oxides by using plasma. A treatment of low-temperature plasma of a molten molybdenum concentrate significantly reduces the contents of S, P, As, Sn, Pb, Zn, Bi, Sb, and C [21].

The considered sources of information show that there are significant results of studies on the kinetics and the mechanism of extracting Mo on the example of individual oxides as well as some types of man-made waste. However, the kinetic regularities and the mechanism of the course of thermal reduction and carbide forming processes in molybdenum oxide concentrates have been studied insufficiently. In them, unlike pure Mo oxides, there are accompanying ore oxide impurities, including Ca, Si, Al, and Mg. They can sig-

nificantly affect the processes of metallization as well as the phase composition and the microstructure of the recovery products. Research in this direction can provide a reduction in the Mo sublimation of oxide compounds during the heat treatment of molybdenum oxide concentrates and the subsequent use of a metallized alloying additive. Proceeding from the above, it is expedient to carry out a comprehensive study of the kinetic regularities of the thermal carbon extraction of a molybdenum oxide concentrate together with researching the phase composition and structure of the corresponding metallization products. The use of raster electron microscopy with an X-ray microanalysis would thus help expand significantly the understanding of the structure and composition of individual sections of the structure in the materials under study.

3. The aim and objectives of the study

The aim of the work is to study the physicochemical characteristics of the thermal carbon extraction of a molybdenum oxide concentrate. This is necessary to determine the parameters that reduce Mo sublimation losses during the processing of ore concentrates and the use of metallized molybdenum-based alloying additives.

To achieve this aim, the following tasks were set to be done:

- to study the kinetic regularities of the thermal carbon extraction of the molybdenum oxide concentrate at different temperatures;
- to investigate the phase composition and the microstructure of the products of extracting the molybdenum oxide concentrate after different heat treatment temperature modes, with respect to the effect on the reduction of the target element loss by sublimation during the alloying process.

4. Materials and methods of studying the properties of products of metallizing the molybdenum oxide concentrate

4.1. Materials and equipment used in the experiment

The source material is a molybdenum oxide concentrate of the brand KMo-2 with a Mo content of at least 55 % by weight (TU 14-5-88-77). Its grinding was carried out on a ball mill with a peripheral unloading of ground material. Sifting was performed through a sieve with a mesh size of 0.45 mm. The extractor was the dust of graphite production. The estimated O/C ratio was 1.33.

Tests on the recovery kinetics were performed on a universal thermogravimetric device of a flow type; the description of the structure and principle of action is given in [4].

The X-ray structural phase analysis of the samples was carried out on the diffractometer DRON-6 (Russia).

Photographs of the microstructure of the samples were obtained on the raster electron microscope REM-106I (Ukraine). The microscope is equipped with a system of X-ray microanalysis to determine the chemical composition of individual sections of the surface of the samples.

4.2. Methods of conducting experiments and determining the properties of the samples

In the kinetic tests, the temperature range was 873–1473 K and the time was 50 minutes. The protective environment was the atmosphere of argon with a linear velocity of gas flow of 3.5×10^{-3} m/s. The mass of the samples under investigation

before the heat treatment was 80 g. During the research, continuous measurement of the mass change of the samples was carried out. For the phase analysis and microstructure tests, samples were selected after the above-mentioned heat treatment at different temperatures.

The phase composition was determined by X-ray diffraction analysis using monochromatic radiation: Cu K α ($\lambda=1.54051$ Å). The measurements were carried out at a tube voltage of $U=40$ kV and an anode current $I=20$ mA. The phase composition was determined using the PDWin 2.0 software package (Russia).

The microstructure of the samples was studied at an accelerating voltage of 20–25 kV and an electron probe current of 52–96 μ A. The working distance to the investigated surface was 10.1–29.8 mm. The composition of the phases was determined without the standard method of calculating the fundamental parameters.

5. Results of studying thermal carbon restoration of the molybdenum oxide concentrate

The graphs (Fig. 1) were approximated by using the least squares method. As a nonlinear regression equation, the following model was chosen:

$$\hat{y} = a + b \ln x, \quad (1)$$

where \hat{y} is the degree of recovery, %; x is time, min; a , b are coefficients of regression, the values of which are summarized in Table 1 for each curve, respectively.

Table 1 shows that the coefficients of correlation r of linearized models are quite close to 1. This means a tight dependence between the indicator and the factor in each case. Table 1 also shows the sum of squares of deviations $\sum e_i^2$.

Since the chosen logarithmic dependence at a point with a zero value of x does not exist, it was not used in the approximation, as can be seen from Fig. 1.

The adequacy of the model is confirmed by the calculated values of Fisher's criterion (Table 1) that exceed the table value $F_{br}=5.32$ at the chosen level of significance $\alpha=5$ %.

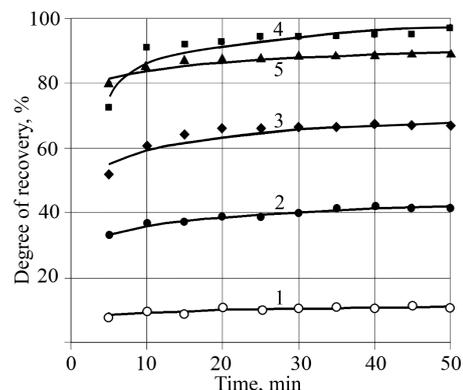


Fig. 1. The kinetics of thermal carbon extraction of the molybdenum oxide concentrate at different temperatures: 1 – 873 K; 2 – 1073 K; 3 – 1223 K; 4 – 1373 K; and 5 – 1473 K

Treatment at a temperature of 873 K provided a recovery rate of 11 % (Fig. 1, curve 1; Fig. 2). A further increase in temperature to 1073 K, 1223 K and 1373 K was characterized by an increase in the degree of recovery to 42 %, 67 %, 88 %, and 95 % respectively.

and 97 %, respectively (Fig. 1, curves 2–4). Treatment at 1473 K provided a higher recovery rate than at 1373 K during the first 5 minutes. (Fig. 1, curve 5). Further, the process proceeded in the diffusion region with a pronounced decrease in the rate of recovery with a degree of 89 %.

Table 1

The combined results of the values of the coefficients of regression a and b for each curve shown in Fig. 1

Curve number	a	b	r	$\sum e_i^2$	F_{kr}
1	6.47	1.15	0.90	1.59	32.48
2	26.72	3.93	0.99	1.33	449.15
3	45.64	5.88	0.92	29.64	45.23
4	66.29	8.16	0.87	107.95	23.86
5	75.52	3.61	0.96	5.48	92.23

Recovery after the treatment at 873 K (Fig. 2) is preferably represented by MoO_2 with the presence of a portion of Mo and Mo_2C . The increase in temperature to 1073 K and 1223 K resulted in an increase in the intensity of revealing Mo and Mo_2C in the recovery products. The process temperature of 1373 K allowed achieving predominance in samples of the Mo phase. A clear manifestation of carbide or oxide phases was not detected at that.

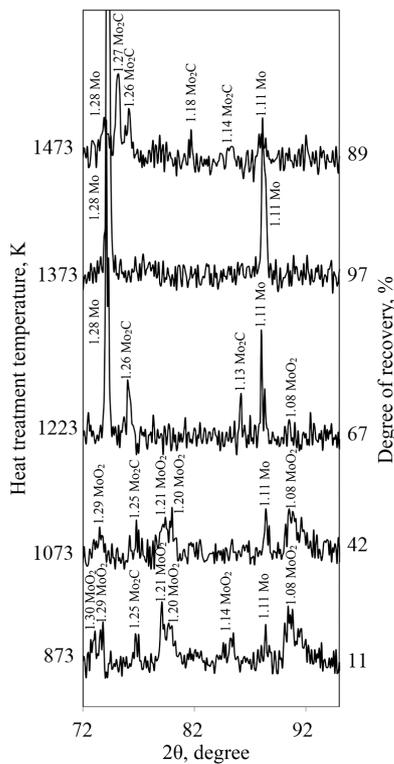


Fig. 2. Plots of diffractograms of samples after heat treatment in accordance with Fig. 1. The values at the diffraction maxima correspond to the parameters of the crystalline lattice

After treatment at 1473 K, a relatively high Mo_2C as to Mo was observed.

The microstructure of the samples is heterogeneous with the presence of particles of different sizes (Fig. 3).

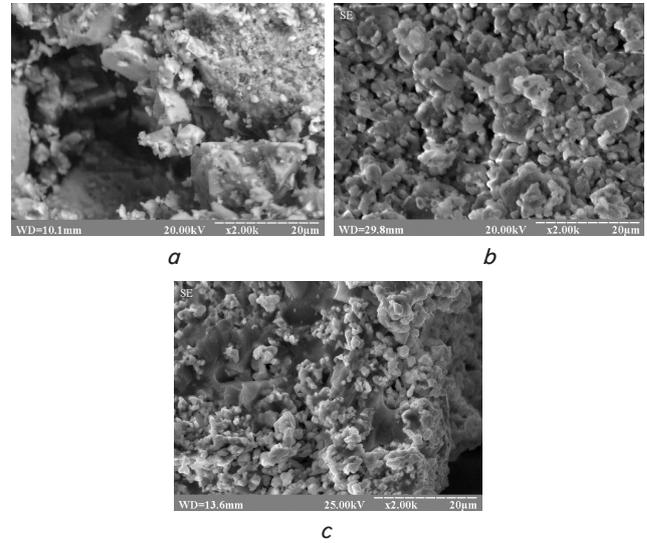


Fig. 3. Photographs of the microstructure of the metallized molybdenum concentrate with an increase of $\times 2000$ after heat treatment at $a - 1073$ K, $b - 1373$ K, and $c - 1473$ K

The oxygen content in the investigated areas was in the range of 6.59–10.34 % by weight; the content of Si as an ore impurity was 0.11 to 1.08 % by weight (Table 2, Fig. 4).

Table 2

The results of the X-ray microanalysis of recovery products according to Fig. 3

Investigated areas	Content of elements, % by weight			Total
	O	Si	Mo	
A	10.34	0.11	89.55	100
B	6.59	0.82	92.59	100
B	7.65	1.08	91.27	100

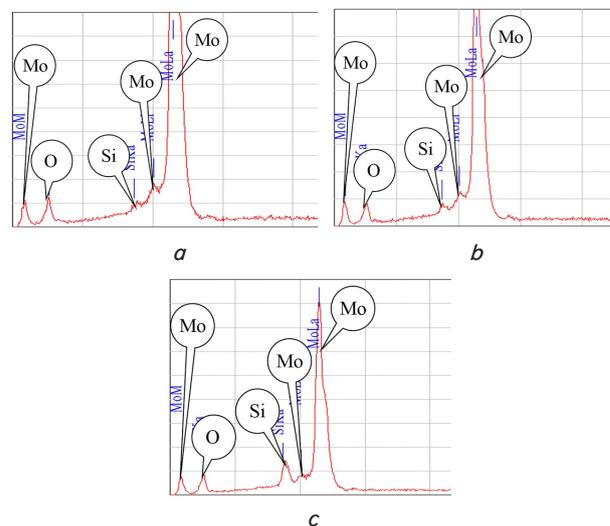


Fig. 4. Spectrographs of the X-ray microanalysis of recovery products according to Fig. 3 after heat treatment at $a - 1073$ K, $b - 1373$ K, and $c - 1473$ K

The study clearly shows an increase in the level of sintering of particles with an increase in the processing temperature from 1073 K to 1473 K. In this case, the recovery at 1473 K

was characterized by the conversion of individual sample sites into an almost continuous sintered layer (Fig. 3, c).

6. Discussion of the results of studying thermal carbon extraction of the molybdenum oxide concentrate

The kinetic tests (Fig. 1) have shown that treatment at 873–1223 K does not provide a sufficiently high recovery (from 11 % to 67 %). Although already at 1223 K there was a significant activation of restorative processes, the heat treatment is of practical interest at 1373 K (the recovery rate of 97 %). This agrees well with the results of [11, 14], where in the same conditions the most effective recovery rates were achieved. An increase in the processing temperature to 1473 K was accompanied by a decrease in the samples' recovery degree down to 89%. This may be due to the formation of superficial "block layers" of sintered particles, which is evidently confirmed by the microscopic tests (Fig. 3). This complicates the gas exchange, which moves the recovery process from the kinetic to the diffusion region. In turn, it raises the relative carbon level in the reaction zone and, as a result, increases the carbidation rate (Fig. 2). Similar tendencies were described in [4], where an excessive temperature increase resulted in a significant increase in the sintering and inhibition of the recovery processes.

It becomes apparent that a further increase in temperature plays a negative role in achieving an increase in the speed and degree of recovery. Also, the above laws are in good agreement with the dynamics of changes in oxygen content in the areas investigated by using X-ray microanalysis (Table 2, Fig. 4).

Therefore, in order to reduce Mo losses, it is necessary to observe heat treatment temperatures that are relatively low but appropriate and sufficient to activate and maintain recovery processes. It is especially important before the transition of most of MoO_3 to lower oxide compounds, having a lower elasticity of vapors by several orders [21]. This corresponds to the research findings of the authors in [8, 11, 20], where Mo_4O_{11} and MoO_2 are intermediate recovery products that can later be converted to Mo or carbide compounds. The detected Mo_2C in the recovery products indicates the parallel occurrence of carbidation and recovery processes. This corresponds to the research findings in [11, 19], from which it follows that it is impossible to obtain a carbon-free product after a thermal carbon restoration.

Based on the above, in terms of the target phase composition, processing is satisfactory in the temperature range of 1223–1473 K. In these conditions, the recovery products mainly consist of Mo and Mo_2C with different ratios. However, taking into account the oxygen recovery rate, the heat treatment is the most beneficial at 1373 K. Under such conditions, the highest recovery rate of 97 % and the almost complete transition of the oxide component to the Mo phase are ensured.

A noteworthy drawback is the lack of research results on the dependence of the phase composition and microstructure

of metallized molybdenum raw materials on the content of the regenerating carbon agent in the charge.

The development of this study is possible in the direction of regulating the chemical and phase composition of metallization products of a mixture of a molybdenum oxide concentrate and man-made alloyed raw materials. This will open the prospect of expanding the parameters of alloying and the branded assortment of alloys for the use of an alloying agent with parallel utilization of man-made waste. Difficulties in trying to develop this study are the lack of a sufficient base of experimental data on the physicochemical patterns of thermal carbon restoration in complex alloyed systems. This indicates the need for additional experiments to determine the interaction of Mo and a complex of alloying elements with a recovery agent and to reduce their losses during heat treatment.

The effectiveness of using a metallized molybdenum concentrate was confirmed by alloying high-speed steel R6M5 in experimental industrial conditions. The melting was carried out in the DSP-18 electric arc furnace on the premises of the Dnipropetsstal PJSC plant (Ukraine). The new alloying agent was used instead of the molybdenum oxide concentrate KMo-2 (TU 14-5-88-77) and the standard ferromolybdenum of the brand FMo60 (GOST 4759-91). The metallized molybdenum concentrate was added under the following technological options:

- 1) into the liquid metal after melting 70–80 % of the charge;
- 2) onto the surface of the metal after the melting of the slag;
- 3) onto the bottom of the furnace under the charge.

The use of the metallized molybdenum concentrate in an average amount of 31 kg/ton has helped achieve an increase in the rate of Mo uptake from 88.9 % to 95.0 % compared to the standard technology. This was achieved by reducing the oxidation potential of the charge and increasing the rate of Mo dissolution in the liquid metal compared with standard ferromolybdenum.

7. Conclusions

1. An increase of the thermal carbon extraction of the molybdenum oxide concentrate from 11 % to 97 % has been determined with an increase in the processing temperature from 873 K to 1373 K, respectively. A further rise in temperature to 1473 K led to a reduction in the degree of recovery to 89 %.

2. The study has found that products of thermal carbon extraction of the molybdenum oxide concentrate after treatment at 873–1073 K consist mainly of MoO_2 with a relatively weak manifestation of Mo and Mo_2C . The treatment at 1223–1473 K provided a predominance of Mo and Mo_2C as to the oxide component. The resulting microstructure was spongy and disordered. Visually, a rise in the level of sintering and the formation of solid layers of sintered particles was observed with an increase in the processing temperature from 1373 K to 1473 K.

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