

Reception Ferrotungsten from Wolframite Concentrate by Alumino-thermic Method

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Abstract

For the smelting ferrotungsten of raw materials (mineral) took us to calculate the charge for each experiment. To optimize the process of obtaining ferrotungsten of wolframite carried out the following activities: to reduce speed and combustion temperature. A change batch positive impact on the development of the combustion process. Experimental results have shown the effectiveness of the introduction of aluminium additives in excess of stoichiometry because the explosive burning regime turned into stationary. In this work the charge calculated on 300 g wolframite concentrate so that as a result of the aluminothermic reaction in the alloy contained 70% of tungsten. Calculations of charge showed that in the case of the smelting of ferro-alloys and master alloys used in this work and have a high melting point (ferrotungsten, ferromolybdenum, etc.) – temperature process of melting alloy is indispensable for the success of melting. To reduce the speed of combustion and the temperature were conducted experiments on selection of ballast additives which allows you to bring the process to a smooth flow of the reaction. As ballast additives used aluminium oxide. It was found that an increase of more than 20 additives reducing the release of the alloy and metal extraction. Also included experiments on selection of warm additives. Use as a warm additive of ammonium nitrate is not desirable because of the rapid reaction and loss of metal slag. The output and the extraction of metal in the application of potassium and sodium nitrate are the same but in the case of potassium nitrate slag can be used as a prolonged potash fertilizer. From the x-ray spectrometer alloys were analyzed.

Introduction

Modern technological process despite use of new kinds of metal products is closely connected with introduction in manufacture of the high-quality special alloyed steels and alloys with new higher operational characteristics that is impossible without manufacture of ferroalloys.

Possessing all kinds of mineral raw materials which is necessary for ferroalloy's manufactures, in economy Republic Kazakhstan there is an obvious prevalence of a raw orientation and stages finishing manufacture are rather poorly developed. In the presence of a rich raw-material base ferroalloy's manufacture is presented only by two large enterprises receiving ferroalloys in the electrooven way.

The state balance stock of tungsten of Republic Kazakhstan as of 1.01.2001 considers 16 deposits, including 10 with balance and 6 with non-commercial stocks.

Since 2004 there was a sharp increase of the prices on molybdenic (7-8 times) and tungsten concentrates (2,5 times). Noted changes: having in Republic Kazakhstan considerable stocks tungsteniferous raw materials working out progressive energy-saving technologies of its reception that is possible with use of SHS-PROCESSES is necessary.

The problem of spent experimental researches consists in an establishment of technological parameters of reception ferrotungsten by means of metallothermic method (SHS) from tungsteniferous raw materials of Republic Kazakhstan (concentrates). Increase in an exit of an alloy and increase in extraction of tungsten at the expense of

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application of the equipment providing superfluous pressure at burning of SHS-SYSTEMS.

Scopes ferrotungsten

Tungsten is widely applied in the modern technics and a number of alloys. 80 % of developed tungsten go on alloy building steels, firm alloys on the basis of tungsten carbide, wearproof and heat resisting alloys.

The considerable share ferrotungsten's concentrates is used in manufacture of special steels [1]. Into structure of widely applied fast-cutting steels enters from 9 to 24 % W; 3,8-4,6 % Cr; 1-5 % V; 4-10 % Co; 0,7-1,5 C. Distinctive feature of a fast-cutting steel consists in its ability self-hardening on air and at a heat of strengthening holiday (700-800°C) thanks to which it keeps high hardness and wear resistance to 600-650°C.

Except fast-cutting other alloyed and tool steels are widely applied: tungsten, chrome-tungstensiliceous, chromewolframic, chrome-tungstenmanganous.

Tungsten is a part of magnetic steels [2]. As the most refractory metal tungsten is a part of some heat resisting alloys.

Raw materials for reception of tungsten and ferrotungsten

The basic raw materials for reception of tungsten both ferrotungsten are scheelite and wolframic concentrates, the maintenance oxide of tungsten and impurity in them STATE VOLUME 213-73 is regulated. The quantity threeoxide of tungsten should be [1] in a concentrate of 55-65 %.

The waste of metal tungsten including ingots, bars, a plate wire etc., is used as secondary raw materials.

Usually their processing is carried out by two ways:

- the first – oxidation scrap material to threeoxide of tungsten in current of air at temperature 1000°C, but losses of tungsten as a result of its evaporation are thus possible. Threeoxide of tungsten dissolve in a solution of alkali and besiege in the form of artificial scheelite [3];
- the second most widespread way of extraction of tungsten from a waste is based on simultaneous oxidation and tungsten dissolution in alkaline nitrates or nitrites. Metal tungsten with huge speed reacts with the fused nitrate sodium with formation wolframate sodium which then is translated in artificial scheelite.

Technology of reception ferrotungsten

The technology of reception out-of-furnace metallothermy in the way in exothermic interaction oxides of concentrate with a reducer. As a reducer can be used carbon, aluminium and ferrosilicon- 75.

The existing technology of reception of ferrotungsten of marks FT-70, FT-65 allows to receive restoration of concentrates by carbon and silicon.

Alloys of other marks melt by silicoaluminum or aluminotermic method.

From thermal balance of melt ferrotungsten [4] follows that at restoration scheelite concentrate exothermic reaction has not enough heat for normal course of process, therefore aluminotermic tungsten from this kind of raw materials melt in the electric furnace.

In England out-of-furnace method melt ferrotungsten from ferberite concentrates [3].

Choice substantiation aluminothermy in the form of SHS-TECHNOLOGY

In the sixtieth years of the XX-th century a number of researchers had been found out the new processes of heterogeneous burning proceeding without participation of oxygen. As fuel in them acted metals (Ta, Nb, Hf, Ti, Zr) and as an oxidizer nonmetals (Si, C, B). During the specified process gaseous products, but only the condensed are not formed. On the basis of these processes the new method of reception of refractory inorganic connections – self-extending high-temperature synthesis has been developed. This method is based on energy use exothermal interactions of components in a burning mode that limits possibilities of SHS-processes.

On a number with this restriction the SHS-way has a number of advantages: simplicity of the equipment, insignificant power expenses, speed of process at the expense of use of energy allocated in the course of reaction, absence of by-products, adverse in the ecological relation. The SHS-technology is used for reception rich alloy, heat resisting alloys and also for welding [5].

SHS-metallurgy used in given work, in particular aluminothermy is a special case metallothermy [6, 7].

Results of last works in area solid-phase burning, in particular aluminotermic burning of oxides in mode SHS, represent potential possibility of change and simplification of commercial technology of reception ferrotungsten. The new

technology has possibility of expansion of a concentration limit of a material from a concentrate.

Calculation and experimental research of methods of an intensification and optimisation of process of melt ferrotungsten by out-of-furnace metallothermy

The melt ferrotungsten from used raw materials (wolframite) demands an individual approach at structure calculation mix materials. It is necessary to consider character of burning easily reconstituted oxides. Presence at structure mix materials calculated stoichiometrical parities of oxides and aluminium leads to a rough current of reaction and metal losses, and also losses occur and at the expense of evaporation tungsten oxide. Following actions were made for optimisation of process of reception ferrotungsten from wolframite: decrease in speed and burning temperature.

Necessary specific warmth of process:

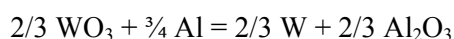
$$\delta H'_{\text{nec}} - 116 \text{ kDzh/g atom}$$

Actual warmth of process at burning 300 g wolframite concentrate $\delta H'_{\text{actual}} = 140,9 \text{ kDzh/g atom}$.

Considering that on heating mix materials on everyone 100°C , specific warmth of process increases by $0,567 \text{ kcal/g atom}$. It is necessary to enter such quantity of ballast for which fusion warmth of process would be spent and the temperature went down. For this purpose it is necessary to spend $3,402 \text{ kDzh/g mol}$ [8].

For selection of conditions of carrying out of process of restoration three oxide tungsten was the model on pure oxide is taken, and as ballast the aluminium powder of mark PA-4, by the size of particles 150 microns was used. Surplus of aluminium in system plays a role flame retardant.

Thermal effect of reaction aluminotermic restoration WO_3 :



Makes $559 \pm 8 \text{ kDzh/mol}$.

For carrying out of experiments samples with the various maintenance of aluminium - from 10 % from stoichiometric composition to 300 % prepared.

At an aluminium lack (-10 %) the role of inhibitor plays reactions threeoxide tungsten which in this case is much.

Structure change mix materials has positively affected development of process of burning. Results of experiments have shown efficiency of introduction of the additive of aluminium overstoichiometry as explosive the burning mode was replaced by the stationary.

Speed of burning is the important parametre therefore the great value is given to its estimation defined by thermopair way. Thermocouple Tungsten-perrhenic took places in apertures of lateral walls of the porcelain not burnt glasses. Thermocouples in diameter of threads 100 microns filled in ceramic straws were connected to a loop oscillograph of mark HO114M. After gathering of installation and charge mix materials in a reactor it was set fire nickel-chrom by a nickel-chrom spiral through the laboratory transformer. Signals from thermocouples were fixed on oscillograph photographic paper.

On the received data speed of burning was defined. Burning time paid off under the formula:

$$\tau = \frac{l}{v} \quad (1)$$

where l – distance between indications of the top and bottom thermocouples, sm;
 v – speed drive photographic papers in an oscillograph, see/second.

Speed of burning was defined from following expression:

$$S = \frac{l_1 \cdot g}{v \cdot \tau} \quad (2)$$

where l_1 - distance between thermocouples;
 g - weight mix materials;
 v - volume mix materials, sm^2 ;
 τ - time, second.

Data of the spent experiments are tabulated. Here results by definition of density of the received metal are yielded. The size of density of metal is the important characteristic of process as it indirectly characterises structure of a metal phase: the its value is closer to tungsten density, the contains impurity in an alloy less.

From table 1 it is visible, that at stoichiometric maintenance of aluminium speed of burning reaches the maximum size. Thus reaction goes with explosive risk, with disorder of melt. At the further increase in aluminium speed falls also process gets quieter character.

Table 1
Speed of burning of aluminum depending on the maintenance
in mix materials aluminum

№	mix materials, g.		Excess quantity of Al over stoichiometrical concentration, %	Burning rate , g/sm ² ·s	Density of alloy, g/sm ³
	Threeoxide of wolfram	Aluminium			
1	250	52,2	-10	16,3	18,8
2	250	58,0	0	18,1	18,9
3	250	63,8	10	17,7	19,0
4	250	69,6	20	17,4	19,1
5	250	87,0	50	16,8	18,9
6	250	116	100	16,1	-
7	250	145	150	6,9	-
8	250	232	300	4,0	-

At speed of burning 3,8-4,2 g/sm²·s reaction products represent sinter without phasedivision. In intervals of a deviation from stoichiometry (-10) - (+100) speed of burning decreases within 10 %. At 150 % it decreases almost in 3 times, and at 300 % - in four.

Thus the alloy density has the maximum size of 19,1 g/sm³ at surplus of aluminium on 20 %. At the further increase in aluminium the alloy density decreases.

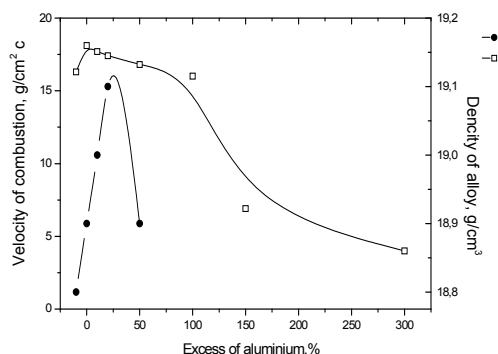


Fig. 1. Dependence of speed of burning and density of an alloy from surplus of aluminium

Influence of aluminium as the ballast additive on parametres of process of burning is especially evident is presented in fig. 1. For a curve characterizing change of density (◻), the harp

maximum testifying to existence of optimum value of a deviation of the maintenance of the additive from stoichiometrical of a parity which excess by adverse image will affect quality of a received product of reaction is observed. For speed of burning (○) boundary conditions of the maintenance of ballast additives less rigid, however, for the coordination of the received results introduction of aluminium as the ballast additive rationally in quantity from 10 to 20 %.

Thus speed of burning 17,4 g/sm²·s and density of a metal phase of 19,1 g/sm³, that to the greatest degree corresponds to pure tungsten (19,3-19,6 g/sm³).

On the basis of the received results of process of burning three oxide of tungsten with aluminium was investigated possibility of reception from wolframite concentrate.

Reception of ferrowolfram from wolframite concentrate by aluminotermic method

The chemical compound industrial ferrotungsten is regulated STATE VOLUME according to which high-percentage ferrotungsten should have a chemical compound resulted in table 2.

Initial material for reception ferrotungsten FW-70 are the concentrates containing in the structure not less of 60-65 % WO₃.

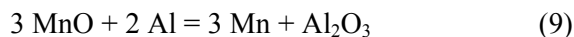
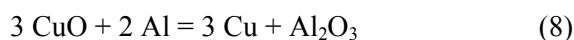
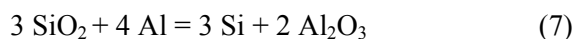
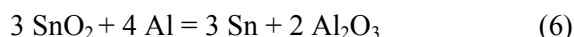
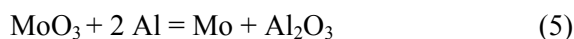
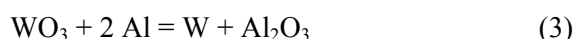
Table 2
The Chemical compound of ferrotungsten

Mark	W, % (less)	The mass maintenance of elements, % (no more)									
		Mo	Mn	Si	C	P	S	Cu	As	Sn	Al
FW 70a	70	7,0	0,3	2,0	0,3	0,06	0,06	0,3	0,08	0,1	6,0

In the given work it was used wolframite concentrate of following structure: WO_3 - 65 %, FeO - 16,47 %, MnO - 7,8 %, SiO_2 - 1,6 %, Cu - 0,12 %, Sn - 0,08 %, MoO_3 - 1,09 %, S - 1,9-2,4 %. For removal of sulphur a concentrate subjected to roasting at 750-780°C.

Structure calculation mix material

Calculation of structure used in experiment mix material according to a standard technique was spent on certain quantity of substance. In the given work mix material paid off on 300 g wolframite concentrate so that as a result of aluminotermic reactions in an alloy contained 70 % of tungsten. Proceeding from concentrate structure in process aluminotermic receptions of ferrotungsten will proceed following exothermic restoration reactions:



Used wolframite concentrate has the size of grain 250-300 microns. From references [11] it is known that the optimum size of particles of a reducer should correspond to dispersion of an oxidizer, therefore for carrying out aluminotermic restoration the aluminium powder of manufacture of Open Company "FLOGA" which has been eliminated to corresponding fraction equal 300 microns was used.

For carrying out of reactions with the maximum effect the necessary quantity of aluminium has been calculated for all used oxides, proceeding from their maintenance in wolframite.

As an example calculation of quantity of aluminium for reaction which was spent as follows is resulted:

In 300gr of wolframite contains – 195 g WO_3 . Molecular weight WO_3 is equal 232 g. On restoration of one molecule WO_3 , according to reaction, 2 molecules of aluminium are required, i.e. on 232 grammes WO_3 should have $2 \cdot 27 = 54$ grammes of aluminium. In 300 grammes wolframite 65 % WO_3 , that is 195 grammes contain. Hence, the necessary quantity of aluminium makes $195 \cdot 54/232 = 45,4$ g.

The necessary quantity of aluminium for all reactions available in this case has been similarly calculated. As a result of the spent calculations it has been received, that the settlement quantity of aluminium at shot of wolframite concentrate 300g makes 68 g. Taking into account that fact, that activity of used aluminium of Open Company "FLOGA" makes 90-92 %, the quantity of aluminium is increased by 10 % and will make 75g. Quantity of a gumboil in which quality fluor-spar was used, undertakes from calculation of 15 % of weight from aluminium.

The quantity of iron which is necessary for adding in mix material for reception of ferrotungsten with the maintenance of 70 % of tungsten, according to the executed calculations and according to reaction, was equaled 12 g.

Proceeding from these calculations and help data specific warmth and temperature of process of melt aluminotermic ferrotungsten for wolframite concentrate have been defined. At course of reactions with participation regenerative oxides containing in wolframite, the following quantity of heat Q in (kcal) is allocated:

$$\text{WO}_3 \rightarrow \text{W} \quad 300 \cdot 0,65 \cdot 865 = 168675$$

$$\text{MoO}_3 \rightarrow \text{Mo} \quad 300 \cdot 0,0109 \cdot 1515 = 4954,0$$

$$\text{SiO}_2 \rightarrow \text{Si} \quad 300 \cdot 0,016 \cdot 973 = 4670,4$$

$$\text{FeO} \rightarrow \text{Fe} \quad 300 \cdot 0,1647 \cdot 956 = 47326$$

$$\text{MnO} \rightarrow \text{Mn} \quad 300 \cdot 0,078 \cdot 875 = 20475$$

$$\text{Total: } Q = 246010 \text{ kcal.}$$

On the basis of these data specific warmth of process under the formula has been calculated:

$$\delta H = \frac{Q}{m} \quad (10)$$

$$\delta H = \frac{246010}{300 + 75 + 12} = 636 \text{ kcal/g or } 2661 \text{ kGzh/g,}$$

where 246010 - quantity of allocated heat,
300 – weight of wolframite concentrate,
75 – weight of aluminium,
12 – iron weight.

For definition of temperature of development of process of burning (T_{pr}) V.A. Bogolyubov's [9] thermal factor which is equal 0,32 was used.

$$T_{pr} = \delta H / 0,32 \quad (11)$$

After carrying out of calculations it has appeared, that the received value of temperature of

process $T_{pr} = 1986^{\circ}\text{C}$ is considerable below temperature measured with the help tungsten – perrhenic thermocouples for the given structure, equal (2450-2500 $^{\circ}\text{C}$) [10, 13]. According to authors this divergence of experimental and settlement data is connected by that Bogolyubov's the used factor is defined on oxide gland and gland does not consider a considerable difference between an average and specific thermal capacity of products of ferrotungsten manufacture and products for aluminotermic restoration oxide.

More precisely it is possible to calculate temperature of regenerative reactions for shot the substances expressed gramme atoms. Taking into account this position and proceeding from quantity of gramme atoms mix material specific warmth of process has been calculated:

$$\delta H' = 140,9 \text{ kDzh/g} \cdot \text{atom.}$$

On the basis of it is possible to calculate more precisely process temperature under the following formula:

$$T_{pr} = 11,6 \cdot \delta H' + 1400 \quad (12)$$

Having substituted in the formula the calculated specific warmth we will find:

$$T_{pr} = 11,6 \cdot 140,9 + 1400 = 3034 \text{ K.}$$

Using literary data [4] it is possible to calculate the warmth of process carried to weight of substance expressed in gramme atoms Q' on the equation:

$$\text{Lg } Q' = \frac{T_{melt}}{3770} + 2,95, \quad (13)$$

where $T_{melt} \text{ oxide} = 1743\text{K}$.

After the spent calculations it has been received, that value $\text{Lg } Q' = 3,412$.

At the same time Q - specific warmth has begun aluminotermic process, carried to 1 kg mix material is equal:

$$Q = 2930 \text{ kdzh/kg.}$$

At such size of a thermal emission of process of burning the system is warmed up to more heat, than fusion temperature of oxide and gland, componentsmix material ($T_{melt} \rightarrow \text{WO}_3 - 1743 \text{ K}$, $\text{FeO} - 1644 \text{ K}$, $\text{MO}_3 - 1068 \text{ K}$, $\text{MnO} - 1923 \text{ K}$, $\text{SiO} - 1986 \text{ K}$, $\text{Fe} - 1802 \text{ K}$) [14].

The carried out calculations have shown that in case of melt of ferroalloys and rich alloys used in the given work and having fusion heats

(ferrotungsten, ferromolybdenous, etc.) - excess of temperature of process over temperature of fusion of an alloy is a necessary condition for successful carrying out of fusion.

Research of influence of concentration of ballasting additives on burning process

For experiment carrying out the calculated components mix material carefully mixed up fallen asleep in a reactor from not burnt porcelain and pressed. Burn mixes it was made нихромовой by a spiral connected to the laboratory transformer. Very rough burning with disorder melt was observed. The alloy exit made the little more than 80 % from the settlement.

For decrease in speed of burning and temperature made experiments on selection of the ballasting additive allowing to lead process to quieter current of reaction. As the ballasting additive used oxide of aluminium as theoretically expected product oxidation-reduction aluminotermic processes.

In table 3 results of the spent experiments are resulted and in fig. 2 influence quantity of a ballast on an alloy exit is shown. From the received data follows that ballast introduction already in number of 10g promotes increase in an exit of an alloy almost on 7,0 %. The highest results are found out at the additive in mix material 20 g of oxide aluminium. Excess of this quantity is not expedient as the exit of a suitable alloy essentially decreases.

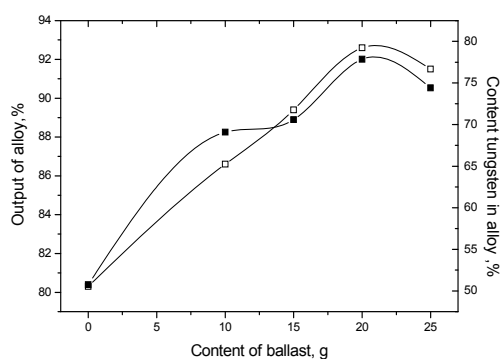


Fig. 2. Influence of quantity of ballasting additive Al_2O_3 on an exit alloy and the tungsten maintenance in the alloy.

In the subsequent experiments as a ballast was used ground slag of the previous swimming trunks. In table 4 results calculations of structure of an alloy and weight of an ingot in comparison with experimentally received data are resulted. The tungsten maintenance in a real ingot and slag was defined by X-ray diffraction technique.

Table 3
Influence of quantity of ballasting additive Al_2O_3 on an alloy exit

№	Structure of mix material, g					Weight of an alloy, g	Settlement weight of an alloy, g	Exit from theory, %
	Concentrate	Fe	Al	Fluor-spar	Oxide aluminium			
1	300	12	75	11	-	174,2	217	80,3
2	300	12	75	11	10	187,0	217	86,6
3	300	12	75	11	15	195,0	217	89,4
4	300	12	75	11	20	200,9	217	92,6
5	300	12	75	11	25	198,5	217	91,5

Table 4
Influences of quantity of a ballast on the tungsten maintenance in an alloy by results of the analysis

№	Makeup mix material, g					Weight of alloy, g	Calculated weight of bullion	Calculated makeup of alloy		Contents of Win slag	Contents of W in alloy, %
	Fe	Concentrate	Al	fluor-spar	scoria			W, %	Fe		
1	12	300	75	11	-	174	217	73	27	2,4	50,75
2	12	300	75	11	10	187	217	73	27	1,89	69,08
3	12	300	75	11	15	195	217	73	27	1,07	70,58
4	12	300	75	11	20	200	217	73	27	0,3	77,86
5	12	300	75	11	25	198	217	73	27	0,9	74,42

The data resulted in table 4 show essential dependence of the maintenance of tungsten in slag and an alloy from quantity entered in mix material ballasting additives. In both cases the experiments put with use of additives give higher indicators of process. At introduction 10g of ballast the tungsten maintenance in slag decreases on 21 % and an alloy increases by 26,5 %. This fact testifies to essential decrease in losses of scarce and expensive metal and economic feasibility of use of ballasting substances. At the same time it has been established

that the additive increase from above 20g conducts to decrease in an exit of an alloy and metal extraction as it is visually shown in table 5. At carrying out of the given experiment it was revealed that in the process of course of regenerative reactions in system there was an essential decrease in temperature of burning mix material. The reason for it is decrease in thermal balance of system at the expense of heat losses on heating and fusion *большого* quantities of a ballast at enough high speed of process.

Table 5
Structures mix material

№	Makeup mix material, %							Exit alloy of theory, %	Contents of wolfram in alloy, %
	Wolfram-containing raw materials	Fe_2O_3	NH_4NO_3	KNO_3	$NaNO_3$	Al	Fluor-spar		
1	58,4	9,2	-	-	-	28,2	4,2	-	-
2	59,2	2,8	17,8	-	-	17,3	2,9	50	32
3	54,55	2,4	-	21,8	-	18,2	3,05	96	36-43
4	54,55	2,4	-	-	21,8	18,2	3,05	93	36-43

The received alloys were analyzed on a x-ray spectrometer. Results of analyses are resulted in table 6.

Table 6
Structure of alloys on X-ray diffraction technique

№	Elements, %									
	Al	Si	Ti	Cr	Mn	Fe	Co	Mo	Cu	W
3	3,18	27,97	2,47	0,18	0,13	17,35	5,19	0,28	0,54	36,61
4	1,57	27,00	2,67	0,23	0,21	16,59	6,32	1,47	0,27	43,27

Analyzing results of experiments have come to conclusion that application as the warming up additive of ammoniac saltpeter is not desirable because of rough course of reaction and metal loss in slag. The exit and metal extraction at application potash and sodium saltpeter are identical, but in case of application potassium saltpeter using results of the previous works slag can be used as the prolonged potash fertilizer.

Conclusions

1. Limits and speed of burning of system WO_3+Al are defined at letting-down mixes by surplus of aluminium (table 1, fig. 1).

2. Thermal effects and temperatures of processes for reception ferrotungsten from a concentrate are calculated.

3. Structures mix material from various raw materials are calculated.

4. Experiments on the calculated structures mix material (tables 3, 5) are made.

5. Influence of the ballasting additive on an exit of an alloy and extraction of target metal (table 4, fig. 2) is revealed.

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