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SULFIDIZATION OF SILVER-POLYMETALLIC ORES OF «GOLTSOVOE» DEPOSIT FOR DECREASING LOSS OF SILVER IN MILL TAILINGS

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The results of laboratory studies of flotation concentration of silver-polymetallic ores of the Goltsovoe deposit at the Omsukchansk concentrator are presented. The results of sieve analysis of mill tailings of the experimental sample of MTP N 101 (N 7577-i) are described. They indicate that a large amount of silver (123 g/t) is lost in the size class – 0.040 mm (yield 50.25 %). According to the results of mineralogical analysis, it is established that the major losses of noble metal are associated with its fine impregnation in oxides, sulfides and silicate rocks. The main silver-bearing minerals are acanthite, polybasite and kustelite (class – 0.040 mm). Experimental studies were carried out in two stages. The purpose of the first stage is to determine the influence of grinding fineness in flotation feed (for a finished class content of 0.074 mm in the range of 60-95 %) for silver recovery at different amounts of butyl potassium xanthate (50, 150, 300 g/t). The purpose of the second stage is to evaluate the effectiveness of sulfidization at different consumption of sodium sulfide $Na_2S \cdot 9H_2O$ (50, 150, 200, 450, 750 g/t – 1 % aqueous solution) under the conditions of the optimal reagent mode established in the first stage of the study. The results of experiments to determine the optimum grinding fineness and studies on the enrichment of silver-polymetallic ore with the use of sodium sulfide as a sulfidizer are presented. The efficiency of the sulfidization process is estimated. The following experimental dependencies of silver recovery are established: on the degree of grinding and consumption of butyl potassium xanthate; on variations of grinding fineness and the consumption of sodium sulphide (with a consumption of butyl xanthate 150 g/t); and on grinding fineness at optimum consumption of sodium sulfide 150 g/t and butyl potassium xanthate 300 g/t. A comparative evaluation of dependence of silver recovery index from the degree of grinding fineness before and after introduction of sodium sulphide (collecting agent consumption of 150 g/t) is given. The mathematical models describing the dependence of silverrecovery on technological parameters, allowing to control the process of flotation of refractory ore with a large number of primary sludges and the tendency of sludging during grinding and concentration (secondary sludges) are given. The optimal consumption of flotation reagents has been experimentally established: sodium sulfide 150 g/t, butyl potassium xanthate 300 g/t with rational grinding (the content of the finished class is 0.074 mm in the flotation feed 85-95 %). Absolute recovery of silver from the ore of the silver-polymetallic deposit «Goltsovoe» in comparison with the technological indicators of the Omsukchansk concentrator processing the material in accordance with the standard mode, increased by 14.1 % (from 70.7 to 84.8 %) with the yield 9.09 % due to intensification of recovery of silver-bearing semi-oxidized sulphides with reduced flotation activity and compensation of high absorptive capacity of fine particles. The amount of silver in the size class – 0.040 mm in the sample MTB N 101 (N 7698-i) after the use of sulfidization was 83 g/t at the yield of 72.01 %, which indicates the efficiency of the process. The losses of silver in mill tailings decreased by 40 g/t (32.52 %). This proves the possibility of processing silver-polymetallic ores of the Goltsovoye deposit without reclaimer operation.

Key words: silver-polymetallic ores, primary and secondary slurries, sodium sulphide, sulfidization, flotation, reagent mode, recovery of fine silver particles, xanthate, grinding fineness

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Introduction. Nowadays the optimization of existing technological processes and search for new, more efficient methods of processing mineral raw materials are required to ensure long-term development of technology for enriching refractory raw materials. At the same time, the technologies being developed should ensure the full use of natural mineral raw materials and meet environmental requirements.

When enriching the silver-polymetallic ores of the Goltsovoe deposit at the Omsukchanskaya concentrator, a significant part of valuable components is lost with the tailings of gravity and flotation concentration. Losses of valuable components are explained by the following reasons: fine dissemination of silver minerals, the complexity of their disintegration during grinding; a significant degree of oxidation of minerals and the need to search for techniques that improve their flotation activity; the presence of a large number of primary sludge and the tendency of ore to slagging during grinding and concentration.

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The solution of the problems associated with refractory ores is reduced to optimizing the grinding regime and processing scheme [2], improving the reagent regime by using both new, often expensive and not easily available collecting agents [1, 3, 4, 8, 13], frothing and depressing reagents, and their compositions with accessible and cheap flotation agents [5, 7, 16]; introduction of pulp ionic composition control systems for automatic control of reagents concentration during processing; introduction of new methods of pulp processing, such as magnetic and electrical processing, bio-heterocoagulation into the enrichment technology; application of effective modern flotation equipment; and separate enrichment of sand and slime fractions [10, 14].

Problem statement. At present, the problem of disintegration of finely dispersed silver enclosed in silicate rock, and consequently the presence of large losses of a noble metal in mill tailings is an unsolved problem for oxidized silver-bearing ores.

The Laboratory of Technological Studies of the JSC «Serebro Magadana» carried out a number of studies (optimization of the grinding regime, improvement of the technological processing scheme, selection of charging material), which improved the technology of ore processing at the Goltsovoe deposit but did not solve the entire set of listed problems related to silver loss from mill tailings (50-60 %).

Sulfidization is the most common method for improving the flotation activity of oxidized silver-containing minerals by adding various reagents, for example sodium sulfide, to the pulp [15, 17]. As a result, the hydrophobicity of the mineral surface is significantly increased [6]. In previous studies it was determined that the absorption capacity of fine particles increases with increasing particle fineness degree [11, 12].

The object of research were silver-polymetallic ores of the Goltsovoe deposit.

The purpose of this work is to evaluate the effectiveness of sulfidization using sodium sulfide additives for flotation concentration of silver-polymetallic ores of the Goltsovoe deposit to reduce losses of valuable components in mill tailings.

Methodology. The study of the material composition of the silver-polymetallic ore of the Goltsovoye deposit. The investigated small technological sample of MPT No. 101 (N 7417-i) with a mass of 50 kg is a material of the ore zone 4 of the silver-polymetallic deposit «Goltsovoe», it was selected by the geological service of JSC «Serebro Magadana» on May 26, 2016.

Silver-polymetallic ores of the «Goltsovoe» deposit – refractory raw materials – are characterized by the following features: a complex mineral composition (Fig.1), a high oxidation degree of 40.3 %, a tendency to slagging – the total content of fine silver minerals in a sieve-class material is –0.5 + 0.074 mm and is estimated to be about 40 % rel., polydisperse impregnation of useful minerals in host rocks and fine dissemination of silver minerals in sulphides, oxides and silicate rock with their mutual intergrowth (the size of silver inclusions varies from 0.002-0.2).

According to the results of the mineralogical analysis of flotation tails, it is established that the main losses of valuable components are related to their fine impregnation in oxides, sulphides and silicate rock. The mineralogical composition is dominated by nonmetallic minerals – quartz, feldspar, chlorite, rhodonite and clay minerals. The content of sulphides is 0.9 % (mainly galena, sphalerite). The main minerals of silver are acanthite and polybasite, in the class – 0.040 mm – kustelite.

A characteristic feature of the «Goltsovoe» deposit is the presence of a large number of pores filled with ocher-clay material (15.5 %). The industrial value of silver-polymetallic ores of the Goltsovoe deposit is silver and lead.

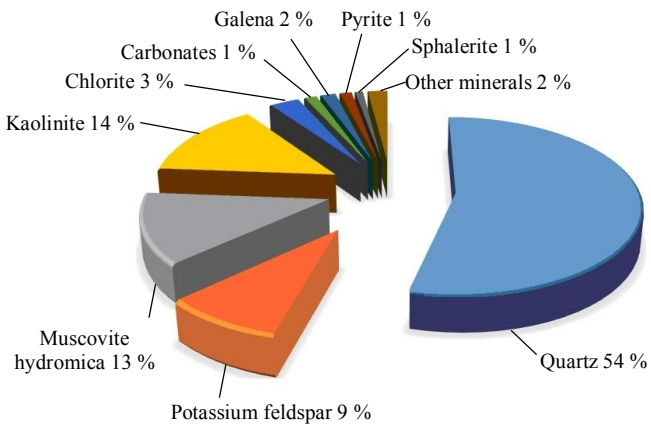

Fig.1. Mineral composition of technological sample N 7417-i

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The mineral composition of the flotation tailings is as follows:

Minerals	Content, %	Minerals	Content, %
Quartz	7	Arsenopyrite	0.9
Field spar	60	Magnetite, rutile, apatite	0.1
Hydromica	25	Limonite	5.5
Biotite	25	Jarosite	5.5
Chlorite	25	Mn oxides	0.4
Ag minerals	0.1	Calamine	0.4
Metallic Sb and As	0.1	Cerussite, anglesite, malachite	0.5
Pyrite, marcasite, galena, sphalerite, chalcopyrite, covellite	0.9	Other secondary minerals	0.5
Fahl ore	0.9	Total	100

The sieve analysis of flotation tailings N 7577-i (Table 1) showed that a significant amount of silver (123 g/t) is in the class – 0.040 mm, the yield of which is 50.25 %.

Table 1

Sieve analysis of flotation tailings No. 7577-i

Grain size class, mm	Yield				Content, g/t		Distribution, %	
	g	%	% by plus	% by minus	Pb	Ag	Pb	Ag
+0.074	124.8	27.17	27.17	100.00	1.35	81.4	15.87	20.92
–0.074 +0.040	103.7	22.58	49.75	72.83	2.45	96.6	23.93	20.63
–0.040	230.8	50.25	100.00	50.25	2.77	123	60.20	58.45
Total	459.3	100	–	–	2.31	105.7	100	100

Discussion of the results. Enrichment of silver-polymetallic ores of the Goltsovoe deposit at the Omsukchansk concentrator is carried out according to the gravity and flotation technology. The pilot studies of flotation concentration were performed on the FM-1M and FM-2M flotation machines (Fig.2).

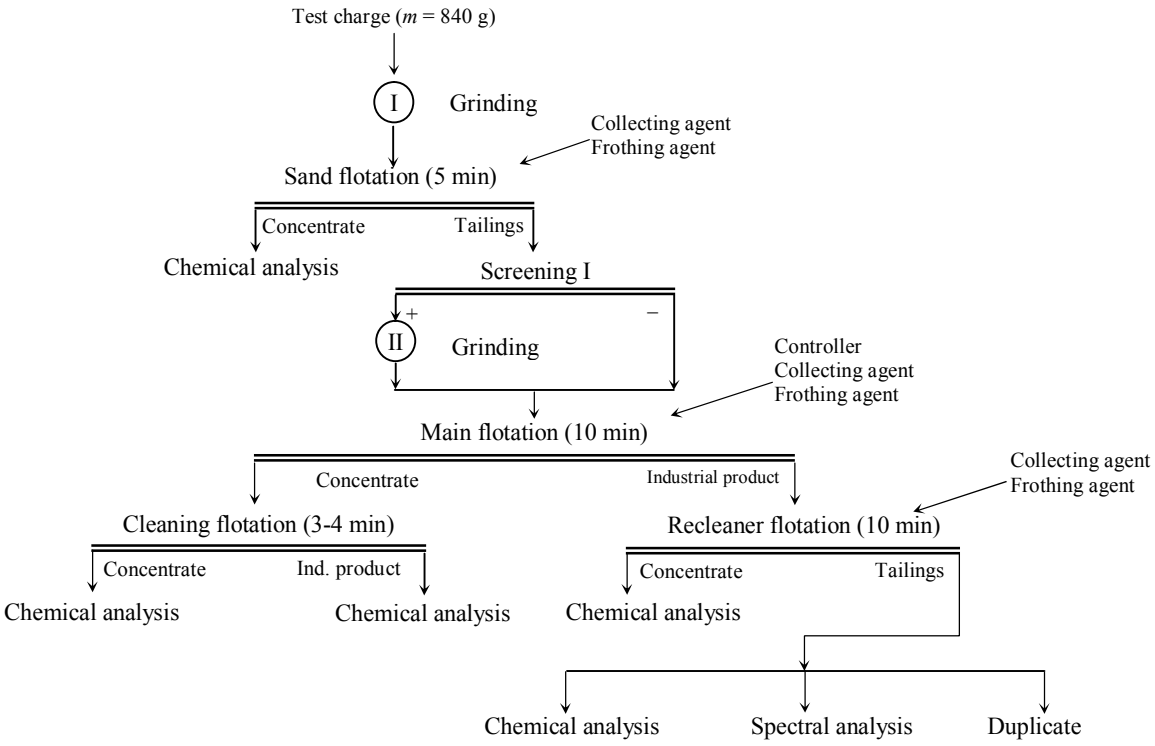

Fig.2. The layout of flotation tests

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The main flotation was carried out in chambers with a volume of 2.0-3.0 liters, the chambers with a capacity of 0.5-1.0 liters were used for cleaning operations.

Reagent regime: the main collecting agent is potassium butyl xanthate (50, 150, 300 g/t – 1 % aqueous solution, feeding points – sand, main and recleaner flotation), the second collector in agent is IMA-I-413 (10 g/t – 1 % aqueous solution, feeding points – sand, main and recleaner flotation), frothing agent – FRIM 2PM (15 g/t – without dilution, feed points – sand, main and recleaner flotation).

Experiments to assess the effectiveness of sulfidization were carried out at different consumption of sodium sulfate $Na_2S{\cdot}9H_2O$ (50, 150, 200, 450, 750 g/t – 1 % aqueous solution, feeding points – main flotation). The sand flotation density is 38-42 % of solids, the main flotation is 25-27 % of solids, the variation change in the finished class content is 0.074 mm, in the sand flotation feed is 45-55 %, the main flotation is 60-95 %.

The results of the experiments were analyzed using X-ray fluorescence, atomic absorption and assay methods of analysis.

The characteristics of the used reagents are shown in Table 2.

Table 2

The characteristics of the used reagents

Reagent type	Reagent	Short description
Collecting	Butyl potassium xanthate C_4H_9OCSSK	Derivatives of coal acids (H_2CO_3). Class of sulfhydryl collectors. It has a collecting ability with respect to native metals
	IMA-I413	Mixture of air floats. Class of sulfhydryl collectors. It has frothing properties, contributes to the extraction of fine classes. The most effective in combination with xanthates
Frothing	FRIM 2PM	It consists of normal and isostructural alcohols with an average molecular mass of 94 ± 14. Close to flotation properties of MIBK
Medium controller	Sodium carbonate Na_2CO_3	White crystals with a density of 2.53 g/cm^3, at 20 ° C are easily soluble in water; pH ≤ 10.5. It is used as a medium controller
Activating	Sodium sulfide $Na_2S{\cdot}9H_2O$	Sulphidizer of oxidized silver-containing minerals of lead and zinc

Studies to determine the optimal consumption of sodium sulfide and associated beneficiation parameters were carried out in several stages.

At the first stage, in order to determine the absorptive capacity of silver minerals in the studied technological sample with respect to the collecting reagent and the effect of the degree of grinding fineness on the silver recover, experiments were carried out with different amounts of butyl potassium xanthate (50, 150, 300 g/t) and the content of the finished class in flotation feeds 60-95 % [9]. As a result of the experiments, the dependencies of silver recovery on the degree of grinding fineness and the consumption of butyl potassium xanthate were obtained (Fig.3).

Statistical analysis of the data made it possible to establish analytical dependencies of the silver recovery in the concentrate from different degrees of grinding fineness:

$$\varepsilon_1 = -0.05x_2 + 6.35x - 162.43;$$
$$\varepsilon_2 = -0.08x_2 + 11.40x - 364.23;$$
$$\varepsilon_3 = -0.05x_2 + 8.60x - 301.9,$$

where $\varepsilon_1 - \varepsilon_3$ is silver recovery at the consumption of butyl potassium xanthate 50; 150; 300 g/t and a different degree of grinding, respectively, %; x is the content of the 0.074 mm class in the flotation feed, %.

The correlation coefficients were: $R_1 = 0.92$; $R_2 = 0.94$; $R_3 = 0.94$, relative measurement error was $\delta_1 = \pm 1.84$ %; $\delta_2 = \pm 1.42$ %; $\delta_3 = \pm 1.73$ %.

Dependences of silver recovery on the degree of grinding fineness at different consumption of butyl xanthate are the following. Dependence at a flow rate of 50 g/t has a regressive character, which indicates a lack of consumption of the collecting agent with high absorptive capacity of flotation minerals, which grows with an increase in the degree of grinding fineness. The maximum silver recovery of 61.8 % was obtained with $\beta_{cl.0.074} = 63\text{-}65$ % in the flotation feed at the collecting agent flow rate of 50 g/t, where $\beta_{cl.0.074}$ is size class content – 0.074 mm.

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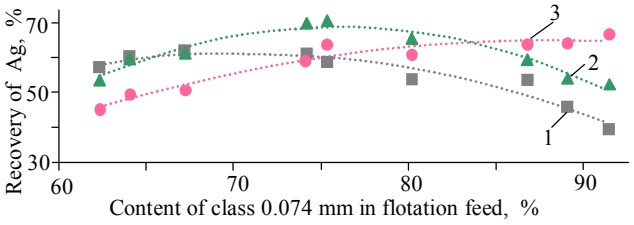

Fig.3. Dependences of silver recovery on grinding fineness and consumption of butyl potassium xanthate
1 – 50 g/t; 2 – 150 g/t; 3 – 300 g/t

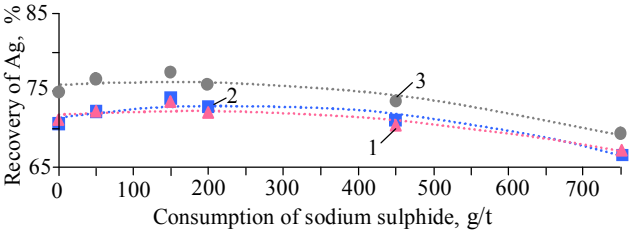

Fig.4. Dependence of silver recovery on grinding fineness and consumption of sodium sulphide
(consumption of butyl potassium xanthate 150 g/t)
1 – 65-75 %; 2 – 75-85 %; 3 – 85-90 %

With an increase in the consumption of butyl potassium xanthate to 300 g/t, the dependence of silver recovery on the degree of grinding fineness acquires a progressive character. The maximum silver recovery of 66.9 % at a collector consumption of 300 g/t was obtained at $\beta_{cl.0.074}$ = 88-90 % in the flotation feed.

The parabolic nature of the dependence of silver recovery on the degree of grinding fineness with the consumption of butyl xanthate 150 g/t indicates the optimum degree of grinding. The maximum recovery of silver at the collecting agent consumption of 150 g/t is achieved with the content of the finished class in the flotation feed 72-75 % and is 70.7 % (yield 6.16 %).

Thus, the conducted studies to determine the absorption capacity of silver minerals in the «Goltsovoe» deposit in relation to butyl potassium xanthate and the effect of grinding on silver recovery have made it possible to establish the following:

1) optimal consumption of butyl potassium xanthate 150 g/t, the degree of crushing 72-75 % of the class – 0.074 mm;

2) increase in grinding fineness (more than 85 %) for recovery of fine-grained silver at this stage does not provide an increase in the silver recovery due to oxidized minerals;

3) increase of collecting agent consumption (300 g/t) for flotation of silver-containing semi-oxidized sulphides with reduced flotation activity at this stage is irrational. Reduction of collecting agent consumption (50 g/t) is inadvisable.

At the second stage, the effect of sodium sulfide consumption on silver recovery was determined at the optimum reagent mode obtained at the first stage of the study. In addition, the influence of the content of the ready-made class in the flotation feed on the effectiveness of sodium sulfide at a collecting agent consumption of 150 g/t has been established.

In the course of the tests, experimental silver recovery curves were obtained showing the change in the absorbance of silver minerals at different consumption of sodium sulfide and the effect of the degree of grinding fineness on the effectiveness of sodium sulfide application (Fig. 4).

Statistical data analysis made it possible to establish analytical dependencies of silver recovery in concentrate from the consumption of sodium sulfide at different degrees of grinding fineness:

$$\varepsilon_1 = 0.01x + 71.50;$$

$$\varepsilon_2 = 0.01x + 71.92;$$

$$\varepsilon_3 = 0.01x + 75.77,$$

where $\varepsilon_1 - \varepsilon_3$ is silver recovery with grinding fineness 65-75; 75-85; 85-95 % relatively; x – consumption of sodium sulfide, g/t.

Coefficients of correlation were: $R_1 = 0.96$; $R_2 = 0.95$; $R_3 = 0.97$, relative measurement error $\delta_1 = \pm 0.92$ %; $\delta_2 = \pm 1.03$ %; $\delta_3 = \pm 0.93$.

Additions of sodium sulphide allow to carry out the flotation at a finer grinding level, which contributes to an increase in silver recovery (by 6.7 %) to 77.4 % with a yield of 7.60 % relative to the recovery of 70.7 % obtained under the standard concentration mode (without sulphidization, $\beta_{cl.0.074}$ = 65-75 % in the flotation feed) (Fig. 5).

Thus, the addition of sodium sulphide, with an increase in the degree of grinding above 85 %, allowed to increase the silver recovery to 77.4 %.

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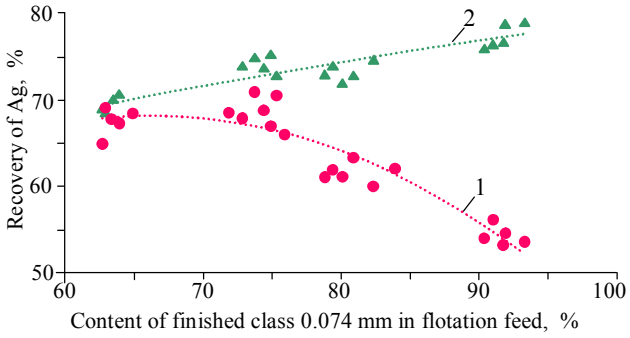

Fig.5. Comparative chart of silver recovery with increase of grinding fineness before and after addition of sodium sulphide and collecting agent consumption 150 g/t
1 – without additives; 2 – with additives

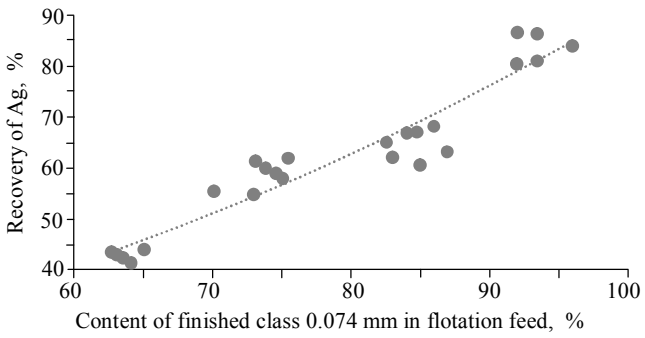

Fig.6. Dependence of silver recovery from grinding fineness at optimal consumption of sodium sulphide 150 g/t and consumption of butyl potassium xanthate 300 g/t

Experiments were carried out with an increased consumption of butyl potassium xanthate to 300 g/t with a previously determined optimal sodium sulphide consumption (150 g/t) and various grinding rates. As a result, the dependence of the change in silver recovery at a consumption of 300 g/t xanthate and 150 g/t sodium sulphide is obtained depending on the content of the finished class in the flotation feed (Fig.6).

The dependence of silver recovery at 150 g/t consumption of sodium sulphide and 300 g/t consumption of collecting agent on the degree of grinding fineness has a progressive character. The best silver recovery rate was obtained with the content of the finished class in the flotation feed above 85 %. Increasing the consumption of the collecting agent is advisable with the addition of sodium sulfide and a high degree of grinding.

The results of the sieve analysis of flotation tailings after application of sulfidization and optimization of the grinding mode are given in Table 3. The amount of silver in the fineness class – 0.040 mm in control sample N 7698-i after the use of sulfidization was 83 g/t at the yield of 63.45 %, therefore, it decreased by 40 g/t.

Table 3

Sieve analysis of flotation tailings N 7698-i after application of sulfidization

Grain size class, mm	Yield				Content, g/t		Distribution, %	
	g	%	% by plus	% by minus	Pb	Ag	Pb	Ag
+0.074	54.1	12.18	12.18	100.00	0.49	50	4.05	8.33
–0.074 + 0.040	108.3	24.38	36.55	87.82	1.11	59	18.38	19.67
–0.040	281.9	63.45	100.00	63.45	1.8	83	77.57	72.00
Total	444.3	100	–	–	1.47	73.1	100	100

A mathematical dependence is obtained

$$\varepsilon = 0.01x^2 - 0.04x + 11.51,$$

where ε – silver recovery at consumption of butyl potassium xanthate 300 g/t, sodium sulfate 150 g/t and various degrees of grinding, %; x – grade content 0.074 mm in flotation feed.

The correlation coefficient is $R = 0.95$ and relative measurement error is $\delta = \pm 1.61$ %.

A significant increase in the yield of the concentrate by 2.93 % relative to the yield obtained in the previous experiments using the reagent mode used in the concentrating plant (without sulphidization) did not contribute to an increase in silver recovery. When carrying out a parallel series of experiments with an increased yield of concentrate, but without additions of sodium sulfide, the recovery of silver was about 70 %.

Regression models obtained experimentally were subjected to the following checks:

– statistical significance of the coefficients of the regression equation;

– general quality of the regression equation.

Thus, the conducted studies on the ore concentration technology for silver-polymetallic deposit «Goltsovoe» allowed to establish the following:

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– the optimum consumption of sodium sulfide is 150 g/t. An increase in the consumption of sodium sulfide (up to 750 g/t) leads to a depression in silver minerals;

– the maximum sodium sulfide efficiency is shown with a high content of the finished class 0.074 mm (over 85 %) in the flotation feed. This is due to the fact that an increase in the degree of grinding fineness allows to recovery fine-dispersed silver contained in oxides, sulphides and silicate rock, which is successfully flotated after sulphidization;

– the optimal consumption of butyl potassium xanthate was 300 g/t;

– the silver recovery rose to 84.8 % with the yield of 9.09 %.

Conclusion. The increase in silver recovery from the ore of the silver-polymetallic deposit «Goltsovoe» was 14.1 % with respect to the extraction of 70.7 % obtained at standard parameters. After sulphidization, silver losses in the flotation tailings with fine classes decreased by 32.52 % due to intensification of recovery of silver-containing semi-oxidized sulfides with reduced flotation capacity and compensation of high absorptive capacity of fine particles.

The obtained data indicate the expediency of sulfidization. However, for a more complete assessment of the effectiveness of the improved reagent mode, an economic evaluation is needed from the point of view of the advisability of increasing the grind and consumption of butyl potassium xanthate to achieve silver recovery of 84.8 %. It is also necessary to carry out an analysis of the suggested reagent mode to determine the effect of increasing grinding on the dehydration performance and the compliance of the granulometric composition of the concentrates obtained with the specifications (TU 201-2015) under this mode.

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