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Research article
Научная статья

Preparation of rhodium concentrate from gold cementate

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Abstract: This article describes studies on improving reprocessing technology gold production cementate (GPC) formed in refining section of chemical metallurgical shop at JSC Uralelektromed, in order to increase the extraction rate of targeted metals into marketable products, diversification of production, achievement of economic effect due to increasing content of precious metals (PM) in individual concentrates. The optimization of GPC reprocessing technology includes intensification of leaching of initial material and filtration of produced pulp, in order to increase the extraction of gold and platinum group metals (PGM) into solution and decrease the circulated PM. This would allow individual products (crude PM) to be obtained with minimum material loss and labor consumption. It is possible to increase rhodium content in concentrate and to reduce its circulation by preliminary oxidating annealing at the temperature above 500 °C. At this temperature hardly soluble trioxide Rh_2O_3 is formed on rhodium surface, insoluble in aqua regia, thus allowing it to deposit in the form of individual product. The influence of temperature and composition of gaseous phase was established upon oxidizing annealing of initial raw stuff ($t = 500 \div 750$ °C) on the composition of rhodium trioxide concentrate ($15 \div 45$ % Rh_2O_3). Reprocessing flowchart of gold production cementate was developed and tested on commercial scale, allowing for the simultaneous production of several products: deposited gold ($Au \geq 98$ %), deposited silver ($Ag \geq 98$ %), PGM concentrate ($Pt \geq 45$ % and $Pd \geq 15$ %), rhodium concentrate ($Rh = 15 \div 45$ %).

Keywords: precipitate of gold recovery, precipitation, precious metals, annealing, leaching, rhodium, concentrate of platinum group metals.

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Получение концентрата родия из цементата золота

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Аннотация: Проведены исследования по совершенствованию технологии переработки цементата производства золота (ЦПЗ), образующегося в аффинажном отделении химико-металлургического цеха АО «Уралэлектромедь», с целью повышения степе-

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ни извлечения целевых металлов в товарные продукты, диверсификации производства, получения экономического эффекта за счет увеличения содержания драгоценных металлов (ДМ) в индивидуальных концентратах. Оптимизация технологии переработки ЦПЗ предусматривает интенсификацию процессов выщелачивания исходного материала и фильтрации полученной пульпы для увеличения показателей извлечения в раствор золота, металлов платиновой группы (МПГ) и снижения находящихся в обороте ДМ, что позволит получить индивидуальные продукты (черновые ДМ) с минимальными материальными затратами и трудоемкостью. Повысить содержание родия в концентрате и сократить его количество в обороте при переработке цементата возможно путем предварительного окислительного обжига при температуре свыше 500 °C, при которой на поверхности родия образуется труднорастворимый триоксид Rh_2O_3, не растворяющийся в «царской водке», что позволяет выделить его в виде индивидуального продукта. Установлено влияние температуры и состава газовой фазы при проведении окислительного обжига исходного сырья (t = 500÷750 °C) на состав концентрата триоксида родия (15÷45 % Rh_2O_3). Разработана и опробована в промышленном варианте схема переработки цементата производства золота, которая позволяет селективно получить несколько продуктов: осажденное золото (Au ≥ 98 %), осажденное серебро (Ag ≥ 98 %), концентрат МПГ (Pt ≥ 45 % и Pd ≥ 15 %), концентрат родия (Rh = 15÷45 %).

Ключевые слова: цементат производства золота, цементация, драгоценные металлы, обжиг, выщелачивание, родий, концентрат металлов платиновой группы.

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Introduction

The content of platinum metals in Ural ores is not high, although at least 97 % of platinoids is extracted from initial raw stuff to anode copper. In the technological circuit of copper production at OAO UMMC, platinoids comprised in crude copper are supplied to AO Uralelektromed. The head enterprise of the company is one of the largest copper refineries in Russia constantly upgrading its technology. In the course of copper electric refining, mud is generated containing precious metals (PM), %: 0.35—0.48 Au, 8.66—11.31 Ag, 0.002—0.004 Pt, 0.025—0.052 Pd, 0.0009—0.0025 Rh [1—3]. Subsequently, PM are concentrated in silver gold alloy (SGA) which is transferred to refining [4—8]. Figure 1 illustrates the technological flowchart of SGA reprocessing at AO Uralelektromed. The products of refining are gold and silver in ingots, as well as platinum group meals concentrate (PGMC) in the form of cathode residue and powder (PGMC-CR and PGMC-P, respectively).

The growing consumption of platinum group metals (PGM) together with increase in their price has led to increasing interest to studies of selective production of refined platinum metals, including the possibility of selective extraction of platinum metals from multicomponent technogenic raw stuff as exemplified by gold production cementate (GPC) [9—13].

The gold production cementate is formed in refining section of chemical metallurgical shop upon PM recovery by metal containing materials from waste solution of recycling gold deposition and PGMC powder, as well as washing waters of dust and gas capturing system of melting stage. The content of main GPC components, % is as follows: 12—30 Au, 2—6 Ag, 14—32 Pt, 5—15 Pd, 2—8 Rh, 25—35 impurities. Impurities in GPC are presented by Cu, Se, Te, Fe and S in amounts up to 10 % each, as well as by other components [14—17].

In order to increase extraction of valuable component and complexity of use of formed process products, GPC is reprocessed both with the collective extraction of impurities and with the selective extraction of precious metals [18; 19]. The composition of the products obtained and efficiency of their extraction depend, in particular, on the nature and properties of solvent [20—22]. The production of individual products of high purity is accompanied by numerous stages of repurification and complexity of the process hardware [23]. AO Uralelektromed currently operates the following flowchart of GPC reprocessing (Fig. 2).

The higher duration (from 4 to 6 h) of cementate dissolution stage should be mentioned. It is directly proportional to the content of non-precious metals: this is accompanied by a decrease in the specifications of direct extraction of metals to targeted products (up to 50 % Au, 40 % Pt, 60 % Pd). It is also interrelated with the long-term filtration of pulp after GPC dissolution in aqua regia solution (from 0.5 to 2 h) due to amorphous structure of the material.

The qualitative phase analysis of the initial cementate is illustrated in Fig. 3. While studying phase structure of the material considered, the possibility of existence of amorphous phases was established (halo with the central point $2\theta \sim 46.89°$) — up to 36 %, stipulated by scatter from unordered phases. The expressed

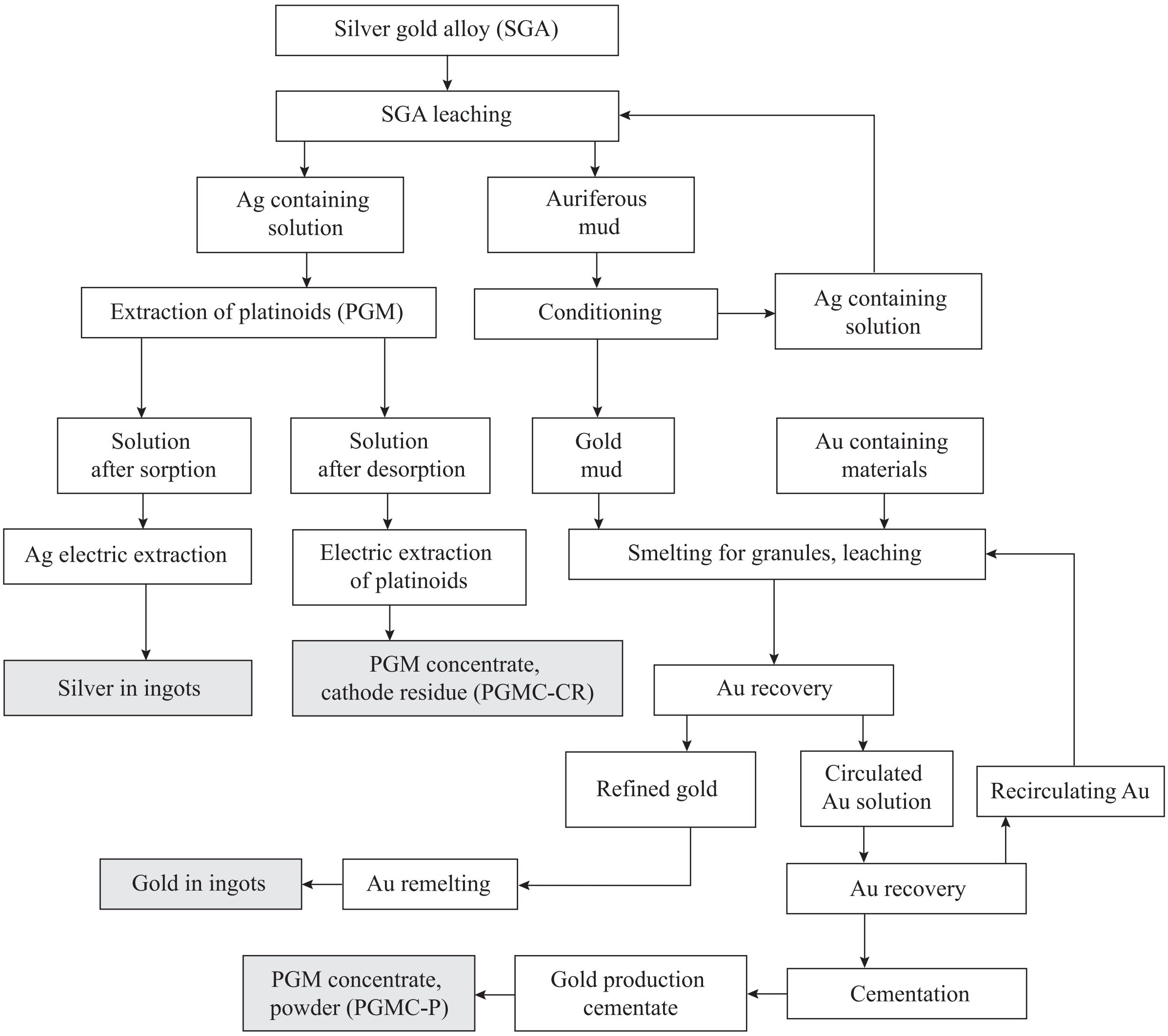

Fig. 1. Flowchart of PM refining at AO Uralelektromed

Рис. 1. Принципиальная схема аффинажа ДМ в АО «Уралэлектромедь»

lines in the diffraction pattern are probably referred to AgAu.

The optimization of GPC reprocessing technology stipulates for oxidizing annealing, leading to intensification of leaching of initial material and filtration of obtained pulp aimed at improving extraction of gold and PGM into solution and decrease in the content of recycling precious metals, thus allowing individual products to be obtained (crude PM), as well as to diversify the production process (production of rhodium concentrate).

According to the existing technology, rhodium is distributed between insoluble residue after GPC leaching (~40 % Rh are extracted) and concentrate of platinoid in the form of powder (PGMC-P, ~60 % Rh) obtained by cementation. Rhodium content in the concentrate is 2—8 %. Rhodium content in PGMC powder can be increased and its recirculating content can be reduced upon GPC reprocessing by preliminary oxidizing annealing at a temperature above 500 °C. At this temperature hardly soluble trioxide Rh_2O_3 is formed on rhodium surface, insoluble in aqua regia, thus allowing it to be deposited in the form of individual product [24—29].

The aim of this work was to improve the reprocessing technology of cementate of gold production including additional production of crude metals (silver: Ag ≥ 98 %, rhodium concentrate: Rh = 15÷45 %) with retention of gold quality (Au ≥ 98 %) and PGM concentrate (Pt ≥ 45 % and Pd ≥ 15 %).

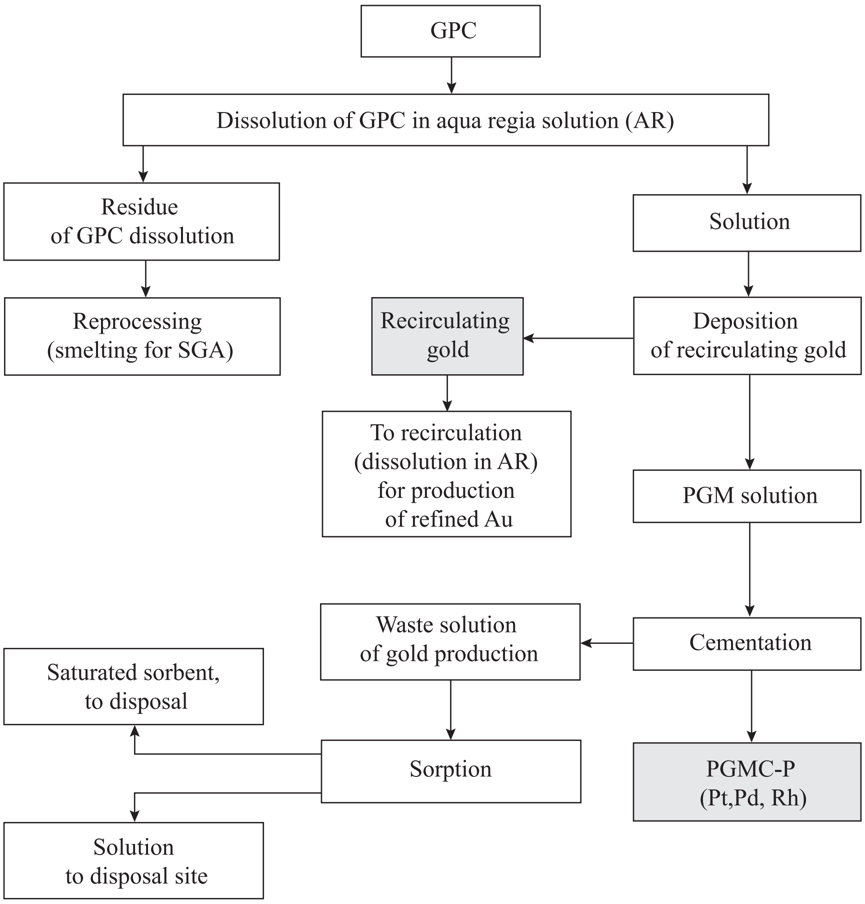

Fig. 2. Existing flowchart of GPC reprocessing

Рис. 2. Принципиальная существующая схема переработки ЦПЗ

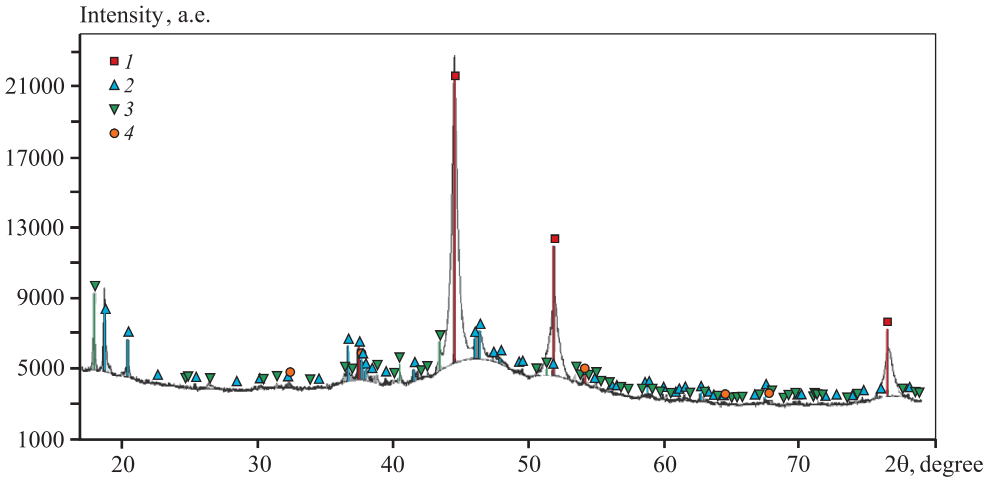

Fig. 3. Qualitative phase analysis of initial GPC
(DiffracSuite EVA v6.0; ICDD PDF-2 2019, diffraction pattern fragment)

1 – AuAg; ***2*** – atacamite $Cu_2Cl(OH)_3$; ***3*** – botallackite $Cu_2(OH)_3Cl$; ***4*** – chlorargyrite AgCl

Рис. 3. Качественный фазовый анализ пробы «ЦПЗ исходный»
(DiffracSuite EVA v6.0; ICDD PDF-2 2019, фрагмент дифрактограммы)

1 – AuAg; ***2*** – атаканит $Cu_2Cl(OH)_3$; ***3*** – боталлакит $Cu_2(OH)_3Cl$; ***4*** – хлораргирит AgCl

Experimental

In order to estimate the possibility of production of concentrate with a higher rhodium content, a series of experiments were carried out according to the improved flowcharts illustrated in Fig. 4.

The experiments differed in the conditions of oxidizing annealing (Table 1). Annealing in test *1* was carried out in a laboratory muffle furnace and compared with the data on reprocessing of initial material (test *1.1*, see Table 2). Tests *2*, *3* were carried out under industrial conditions: electric furnace of indirect heating; material layer height not more than 2 cm. The parameters of other technological procedures were identical.

GPC leaching after annealing was carried out in laboratory and commercial reactors at 85—95 °C and L : S = 5 mixture of acids with the following composition, vol.% : 85—95 HCl, 5—15 HNO_3.

The chemical analysis of process products was carried out using an inductively coupled Spectroblue plasma atomic emission spectrometer (Spectro Analytical Instruments, Germany). The final products were analyzed by atomic emission spectroscopy with spark emission of spectrum using a Spectrolab M12 (Germany) emission spectrometer.

Samples of the cementate and products of its annealing were also studied by *X*-ray phase analysis (XPA) using a Bruker D8 Advance diffractometer (Bruker Corp., USA) in the range of diffraction angles $2\theta = 15\div80°$ in CoK_α-radiation with the increment of 0.025° using a LynxEye position sensitive detector. The total time of recording was 7.5 h, the equivalent time per step: 1860 s. The qualitative phase analysis was carried out using Bruker DiffracSuite EVA v6.0 software and database of reference diffraction spectra ICDD PDF-2 (v. 2019). The quantitative phase analysis was carried out in the Bruker TOPAS 5.0 full profile package according to Rietveld.The crystalline phases were taken from Crystallography Open Database (Cambridge university, Great Britain). Micro images of the

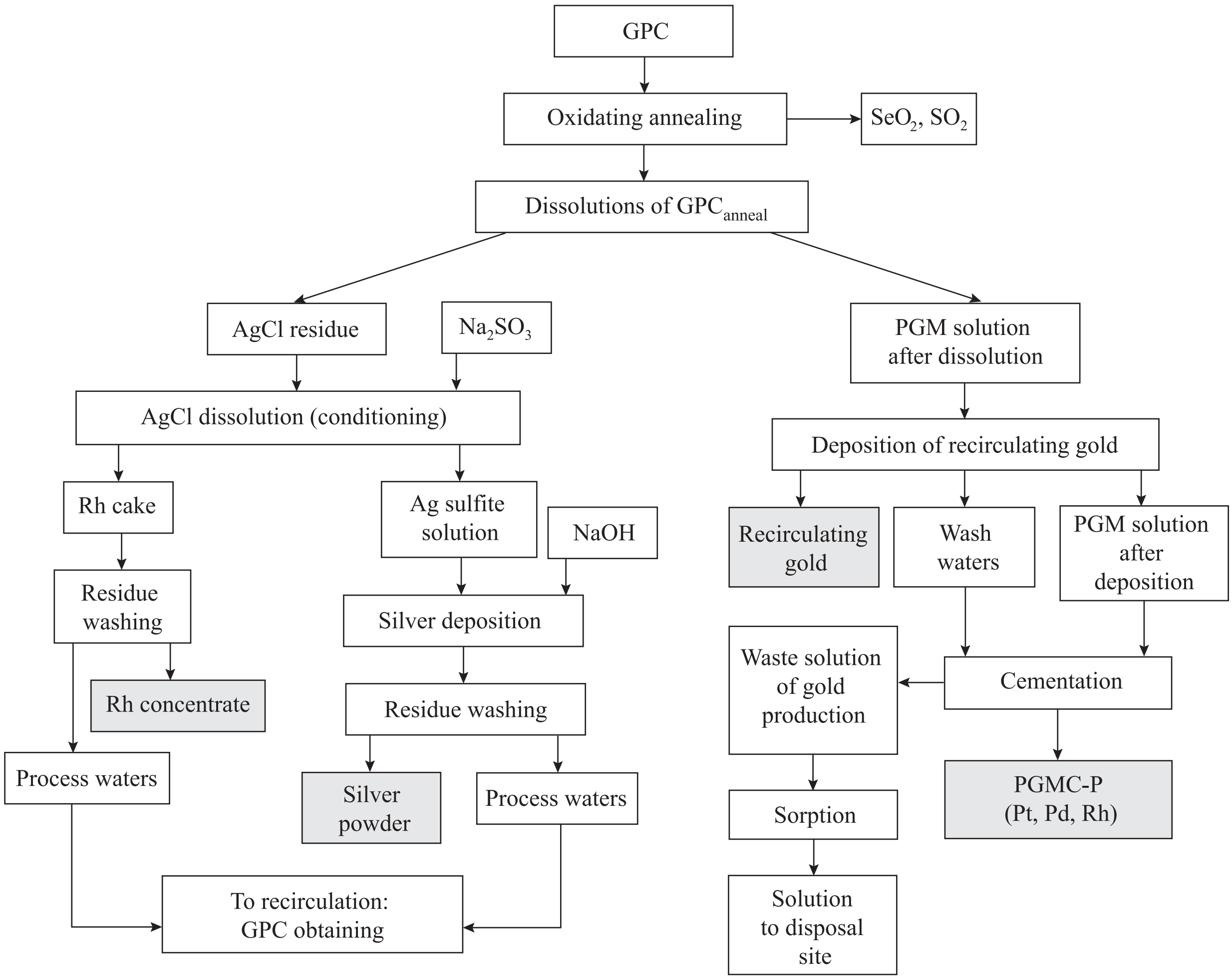

Fig. 4. Proposed flowchart of GPC reprocessing

Рис. 4. Предлагаемая технологическая схема переработки ЦПЗ

Table 1. **Technological parameters of GPC annealing**

Таблица 1. Технологические параметры обжига ЦПЗ

Test No.	*t*, °C	Duration, h	GPC weight, kg	Annealing conditions
1.1	–	–	0.2	Laboratory, w/o preliminary annealing
1	750	10–12	0.2	Laboratory, periodical air supply (0.05 n.m^3/h)
2	500	8–10	28.1	Industrial, pure oxygen supply (20 n.m^3/h)
3	620	24–30	20.7	Industrial, periodical air supply (5 n.m^3/h)
4	720	8–10	25.6	Industrial, pure oxygen supply (20 n.m^3/h)
5	720	12–14	21.5	Industrial, periodical air supply Industrial (5 n.m^3/h)

samples were obtained using a Tescan Vega electron microscope (Tescan, Czech Republic) with the magnification of 60$^\times$ and 400$^\times$.

Results and discussion

In the course of leaching of annealed materials both the duration of material dissolution and filtration duration of the pulps (Fig. 5) decrease by 50—70 %, related with variations in residue structure (Fig. 6, laboratory experiments). Despite the additional procedure of annealing with the duration of 8—16 h, the technology does not result in increase of unfinished production. This allows the number of repeated dissolutions to be reduced and up to 5 % of rhodium from circulation to be removed. Table 2 summarizes the leaching degrees of metals into solution in each experiment.

The qualitative phase analysis of GPC after annealing is illustrated in Fig. 6. Variations in the residue structure are observed, as evidenced by XPA data. According to the diffraction pattern, the formation of crystals was identified, and the fraction of amorphous phases significantly decreased. The results of *X*-ray spectral microanalysis also confirm the conclusions obtained. In comparison with the initial cementate (Fig. 7, *a*), the product after annealing (Fig. 7, *b*) demonstrates obviously distinguishable Mcalpineite crystals Cu_3TeO_6 formed as a result of oxidizing annealing. The possibility of crystal formation was demonstrated in [30]. Additional confirmation of the conclusions about variations of residue structure are the practical results of cementate filtrate rates, before and after annealing.

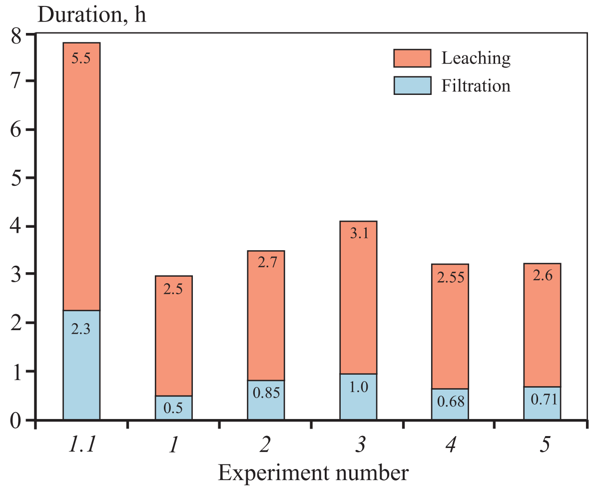

Fig. 5. Duration of leaching and filtration stages as a function of conditions of preliminary annealing

Рис. 5. Зависимость продолжительности стадий выщелачивания и фильтрации от условий предварительного обжига

Table 2. **Degree of metal leaching into solution, %**

Таблица 2. Степень выщелачивания металлов в раствор, %

Test No.	Au	Ag	Pt	Pd	Rh
1.1	80	9	77	74	62
1	99.99	4	86	82	30
2	99.99	3	99.99	99.99	50
3	99.99	1.5	88	72	34
4	99.99	2.6	95.7	86.2	32
5	99.99	1.8	96.4	90.1	33

Neither the composition of gaseous phase (oxygen, air, or oxygen air mixture (OAM)), nor the mode of gas supply (0,05—20 n.m^3/h) exerted noticeable impact on the efficiency of rhodium oxidation, due to excessive content of oxidizer. The gas composition in commercial tests was stipulated by the technological features and parameters of the annealing furnaces involved. The efficiency of rhodium oxidation was influenced to the highest extent by the temperature: at $t = 750$ °C only 30 % Rh is dissolved. The significant difference in the degrees of PM extraction into solution is stipulated not only by the annealing temperature, but also by various content of metallic impurities (Cu, Te, Fe, Sb) in different batches of GPC, their influence will be estimated in further studies.

Excessive nitric acid was removed from the leaching solution by the addition of ethyl alcohol solution upon heating. Table 3 summarizes the compositions of recirculating gold and PGMC. The PM extraction (Au, Pt, Pd, Rh) from leaching solution to final products was more than 99 %.

The insoluble residue of GPC dissolution after an-

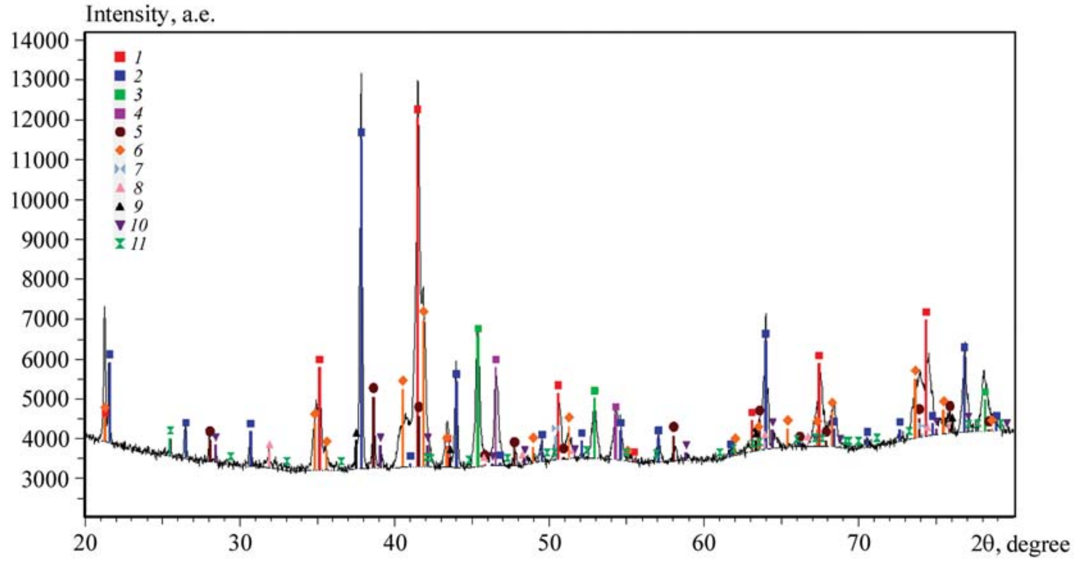

Fig. 6. Qualitative phase analysis of GPC after annealing
(DiffracSuite EVA v6.0; ICDD PDF-2 2019, diffraction pattern fragment)

Phases: ***1*** – iron manganate Fe_2MnO_4; ***2*** – copper nickel tellurium oxide $Cu_2Ni(TeO_6)$; ***3*** – $Au_{0.8}Cu_{0.2}$; ***4*** – platinum Pt; ***5*** – hematite Fe_2O_3; ***6*** – copper ferrite $CuFe_2O_4$; ***7*** – propatagium $Fe_{0.75}Pt_{0.25}$; ***8*** – rutile TiO_2; ***9*** – AgO; ***10*** – $(Cr_{0.88}Ti_{0.12})_2O_3$; ***11*** – cristobalite SiO_2

Рис. 6. Качественный фазовый анализ пробы «ЦПЗ после обжига»
(DiffracSuite EVA v6.0; ICDD PDF-2 2019, фрагмент дифрактограммы)

Фазы: ***1*** – манганат железа Fe_2MnO_4; ***2*** – медно-никелевый оксид теллура $Cu_2Ni(TeO_6)$; ***3*** – $Au_{0,8}Cu_{0,2}$; ***4*** – платина Pt; ***5*** – гематит Fe_2O_3; ***6*** – феррит меди $CuFe_2O_4$; ***7*** – ферроплатина $Fe_{0,75}Pt_{0,25}$; ***8*** – рутил TiO_2; ***9*** – AgO; ***10*** – $(Cr_{0,88}Ti_{0,12})_2O_3$; ***11*** – кристобалит SiO_2

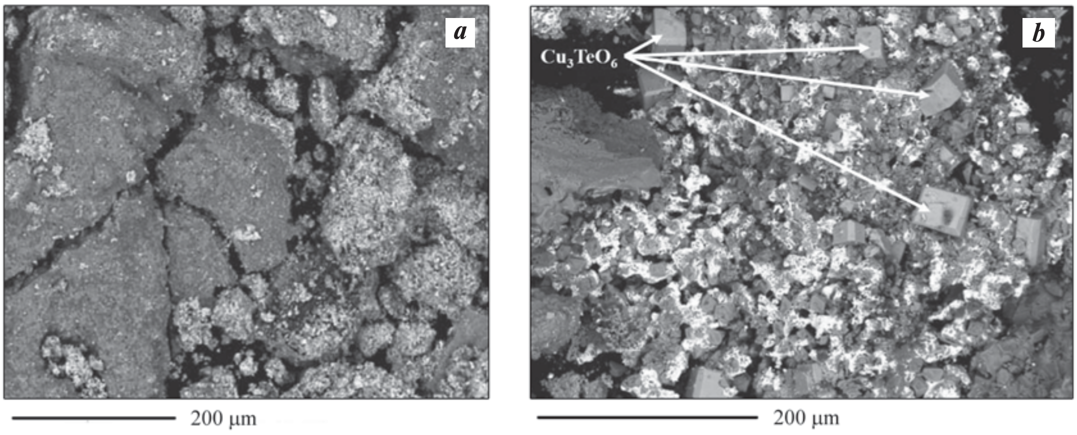

Fig. 7. GPC micro images before (***a***) and after (***b***) annealing

Рис. 7. Микрофотографии ЦПЗ до (***a***) и после (***b***) обжига

Table 3. **PM content (%) in recirculated gold and PGMC**

Таблица 3. Содержание ДМ (%) в золоте оборотном и КМПГ

Test No.	Au	Ag	Pt	Pd	Rh
1.1	99.6/0.46	0.022/1.01	0.017/32.53	0.029/11.5	0.045/9.12
1	99.3/–	0.020/0.005	0.178/46.15	0.199/22.70	0.001/2.10
2	98.9/0.012	0.257/0.720	0.234/38.80	0.010/15.12	0.010/3.10
3	99.4/0.056	0.015/0.329	0.045/48.13	0.105/19.79	0.000/2.52
4	98.6/0.021	0.153/0.56	0.184/45.7	0.095/20.1	0.011/2.15
5	99.01/0.015	0.104/0.43	0.078/44.5	0.102/19.97	0.009/2,42

Remark: numerator – recirculated gold, denominator – PGMC.

Table 4. **PM content in rhodium concentrate, %**

Таблица 4. Содержание ДМ в концентрате родия, %

Test No.	Rh	Au	Ag	Pt	Pd
1.1	8.4	0.1	0.15	3.5	2.8
1	45.00	0.15	0.250	1.17	0.58
2	20.08	1.07	0.543	3.96	6.16
3	15.10	0.54	5.200	1.600	11.7
4	40.25	0.96	1.2	1.512	8.92
5	37.89	0.85	1.08	2.08	9.07

Table 5. **PM content in silver residue, %**

Таблица 5. Содержание ДМ в осадке серебра, %

Test No.	Au	Ag	Pt	Pd	Rh
1.1	0.05	85.1	2.3	1.58	0.45
1	–	99.59	0.00	0.00	0.00
2	0.18	94.61	0.77	0.15	0.30
3	2.34	86.80	1.70	0.68	0.39
4	0.26	92.48	0.89	0.57	0.05
5	0.34	93.26	1.04	0.39	0.12

nealing in aqua regia is comprised mainly of silver chloride (AgCl) and rhodium oxide (Rh_2O_3). An increase in rhodium content in solid phase was achieved by leaching of insoluble residue by sodium sulfite. The silver in testes *1*/*2*/*3* passes into solution with the following concentrations, g/dm^3: 9.18/9.2/7.9. The silver extraction is, %: 99.99/99.99/75. The final solid residue is rhodium concentrate (Table 4), suitable for subsequent reprocessing with production of metallic rhodium.

From the sulfite conditioning solutions obtained, the silver was reduced to metallic state by the addition of caustic soda:

$$2AgCl + Na_2SO_3 + 2NaOH \rightarrow$$
$$\rightarrow 2Ag\downarrow + Na_2SO_4 + 2NaCl + H_2O. \qquad (1)$$

The analysis results of silver residue are summarized in Table 5.

The annealing conditions and rhodium contents in the products of various stages are illustrated in Fig. 8 and Table 6.

The efficiency of rhodium oxidation directly depends on the annealing temperature mode. An increase

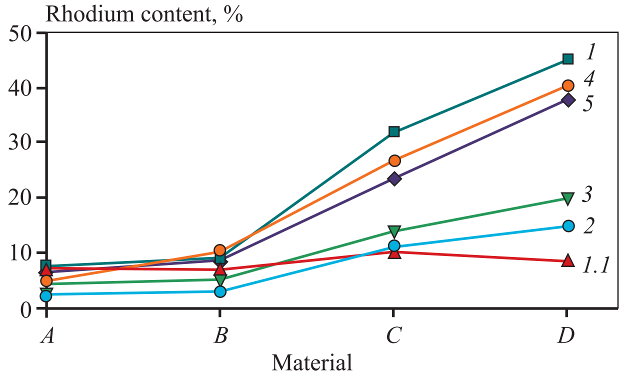

Fig. 8. Rhodium content in GPC reprocessing products annealed under various conditions

Material: ***A*** – initial GPC; ***B*** – GPC after annealing; ***C*** – insoluble residue of GPC leaching; ***D*** – rhodium concentrate
t, °C: ***1.1*** – w/o annealing; ***1*** – 750, OAM; ***2*** – 500, O_2; ***3*** – 620, OAM; ***4*** – 720, O_2; ***5*** – 720, OAM

Рис. 8. Зависимость содержания родия в продуктах переработки ЦПЗ, обожженного при различных условиях

Материал: ***А*** – ЦПЗ исходный; ***В*** – ЦПЗ после обжига; ***С*** – нерастворимый остаток выщелачивания ЦПЗ; ***D*** – концентрат родия
t, °С: ***1.1*** – без обжига; ***1*** – 750, КВС; ***2*** – 500, O_2; ***3*** – 620, КВС; ***4*** – 720, O_2; ***5*** – 720, КВС

Table 6. **Rhodium concentration in initial cementate and products of its reprocessing, %**

Таблица 6. Концентрация родия в исходном цементате и продуктах его переработки, %

Test No.	Annealing conditions		Initial cementate	Cementate after annealing	Residue of AR dissolution*	Rhodium concentrate**
	t, °C	Gas				
1.1	W/o annealing		7.00	7.00	10.29	8.40
1	750	OAM	7.00	9.30	31.95	45.00
2	500	O_2	2.33	2.95	11.00	15.00
3	620	OAM	4.40	5.20	14.00	20.00
4	720	O_2	5.07	10.27	26.83	40.25
5	720	OAM	6.52	8.68	23.72	37.89

* Material residue after dissolution in aqua regia.

** The obtained rhodium concentrate after performed procedures according to flowchart in Fig. 4.

in the annealing temperature from 500 to 750 °C increases the rhodium content in the concentrate from 15 to 45 %.

Conclusions

The reprocessing flowchart of collective cementate of gold production containing precious metal at AO Uralelektromed was developed and tested. The dependence of the extent of rhodium oxidation on the temperature of preliminary annealing of the cementate was established. Its increase from 500 to 750 °C enhances the content of the targeted metal in the marketable product (concentrate) from 15 to 45 % Rh. The optimum parameters of preliminary oxidating annealing are as follows: 750 °C, 2 hours.

The proposed technological flowchart of GPC reprocessing allows the following individual products to be achieved: silver (Ag ≥ 98 %) and rhodium concentrate (Rh = 15÷45 %) while preserving the quality of gold (Au ≥ 98 %) and PGM concentrate (Pt ≥ 45 %, Pd ≥ 15 %).

In order to further develop this topic, the refinement of rhodium concentrate, adjustment of optimum conditions of its reprocessing and commercial implementation of the technology, could be additionally studied.

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