

SNF processing electrochemical operations: liquid-metal and salt medium purification^{*}

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Abstract

The paper investigates the process of regeneration of a liquid metal medium used in the pyroelectrochemical reprocessing of spent mixed uranium-plutonium nitride fuel produced by a fast neutron reactor. The investigation concerns the interaction of liquid cadmium with sludge formed during the anodic dissolution of ceramic nitride pellets in a 3LiCl-2KCl melt medium as well as the possibility of its purification by filtration from individual metal fission products. Anode sludge is represented by fission products of the platinum group, zirconium, molybdenum and technetium. It was determined by scanning electron microscopy that the metal product is composed of several intergrowth phases. It was found that upon contact of a polymetallic alloy simulating anode sludge with a melt, the liquid metal phase is saturated to 0.025 wt% of Pd, 0.01 wt% of Rh for 50 hours at 500 °C, while zirconium forms an insoluble dispersed intermetallic compound ZrCd₃. Powders of molybdenum and technetium, which are not wetted with cadmium, can be completely removed using a filter mesh of plain weaving of the P-200 type. It is also possible to remove zirconium from anodic cadmium by filtration. The filtration efficiency of ruthenium and palladium powders did not exceed 54.3 and 13.1 wt%, respectively, due to partial dissolution and thinning of particles, which will lead to saturation of the liquid metal phase and the need to purify it by alternative methods.

Keywords

Anode sludge, PFC, filter element, 3LiCl-2KCl, electrolyzer, SNF, intermetallide

Introduction

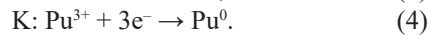
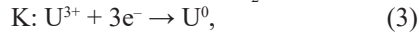
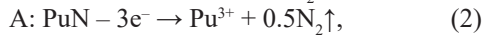
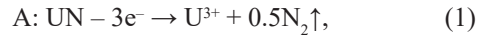
Within the framework of the Russian program for the accelerated nuclear power development focused on the closed nuclear fuel cycle (CNFC), a pilot demonstration power complex is being created, which includes a power unit with a fast neutron reactor BREST-OD-300, a fuel fabrication-refabrication module, and a reprocessing and radioactive waste (RW) management module (Adamov et al. 2015, Aleksakhin et al. 2016). It is planned to reprocess

spent nuclear fuel (SNF) with a high burnup and a short holding time according to a combined process scheme, including pyroelectrochemical and hydrometallurgical processes (Shadrin et al. 2014, 2016, Goff 2008).

At the early stages of development, it was assumed that one of the main operations would be electrorefining of 3LiCl-2KCl salts in the eutectic melt at a temperature of 450–500 °C (Lineberry et al. 1993, Brunsvold et al. 2000, Koyama et al. 2007, Westphal et al. 2007, Goff 2008, Westphal et al. 2008, Goff et al. 2011, Shadrin et al. 2014, 2016, Osipenko

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et al. 2018, Prototype Cathode Processor 2021). Pellets of mixed nitride uranium-plutonium (MNUP) SNF were subjected to electrochemical dissolution on a liquid-metal cadmium cathode. The target components, represented by actinides, passed into the melt in the form of chlorides, and then were reduced at the cathode with the formation of a collective metal product according to the equations:



Alkaline, alkaline-earth and rare-earth metals saturated the electrolyte. Noble and “semi-noble” fission products (FPs), including molybdenum, technetium, ruthenium, rhodium, and palladium, according to thermodynamic calculations, were supposed to form anode sludge, i.e., an insoluble dispersed metal phase that accumulated in liquid cadmium. As the content of individual isotopes increased, their heat release could have led to an undesirable change in the parameters of the electrochemical process, overheating, and boiling up of the liquid metal anode. As the main solution, it was proposed to develop a filtration technology, in which the contents of the electrolyzer, including the metal and salt phases, were passed through a phase separating partition. Melt filtration-based refining methods are widely used in metallurgical production for purifying metals and making cast products from them (Sidorov et al. 2015, Babashov et al. 2020).

It should be noted that in the SNF pyroelectrochemical reprocessing technology, the process of electrorefining was excluded from the process scheme due to the impossibility of providing the required performance of a single unit. When the required current density at the anode was set, the SNF pellets were covered with insoluble current-insulating films consisting of compounds of the U_2N_3 and UNCl type (Zhitkov et al. 2020), which prevented the normal course of the process. Subsequently, the electrochemical processing was replaced by the process of ‘soft’ chlorination with a CdCl_2 - LiCl - KCl melt (Osipenko 2016, 2017, Potapov et al. 2018, Zhitkov et al. 2020).

Nevertheless, the study of the filtration cleaning process may be of interest to related industries that use liquid metal and molten salt media in their production practice.

The purpose of this paper is to study the process of cadmium purification from anode sludge. In the course of the work, the authors studied the properties of the anode sludge simulator and its stability in the cadmium melt, synchronous filtration purification of cadmium and salt electrolyte from inactive components of the anode sludge simulator.

Equipment and materials

Synthesis of inactive samples simulating anode sludge was carried out in a 5 SA electric arc furnace (Centorr Vacuum Industries, USA). Rhenium was used as a technetium simulator, the similarity of the chemical properties of which is due to the proximity of the atomic radius due to the lanthanide contraction of the electron shell (Tananaev et al. 2006). Due to the uncertainty of the behavior of zirconium in the considered metallic system, studies were carried out on two alloys: (1) with and (2) without the added element. The compositions are presented in Table 1.

Table 1. Composition of the studied samples of anode sludge simulators

Sample	Content of elements, wt.%					
	Re	Mo	Zr	Ru	Rh	Pd
Alloy-1	6.1	24.1	20.1	24.1	7.1	18.5
Alloy-2	7.7	30.1	0.0	30.1	8.9	23.1

Filtration purification experiments were carried out on a laboratory facility with an inert atmosphere, which excludes the interaction of the 3LiCl - 2KCl electrolyte with atmospheric oxygen and water vapor. Inside the volume filled with HP (high purity) argon, two vertical resistance furnaces were placed, in one of them a quartz tube with an alundum crucible for the melt was installed, while the other contained a tube with a fixed filter and a receiving crucible in the lower part. Vacuum was supplied to the furnace with the filter. The diagram of the laboratory facility is shown in Fig. 1.

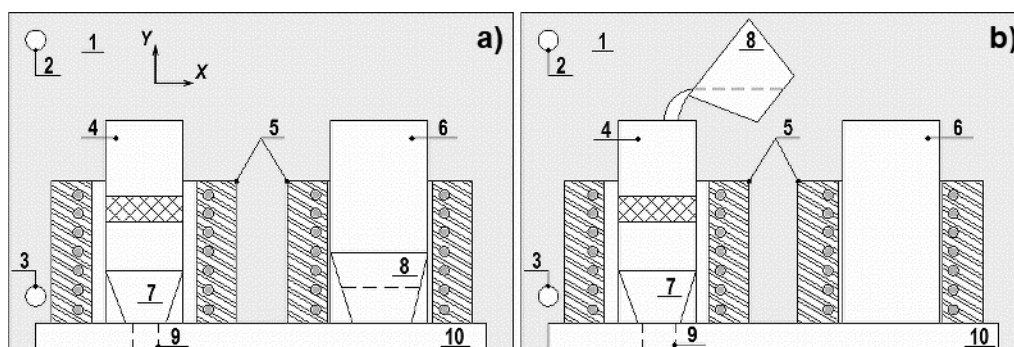


Figure 1. Diagram of the laboratory facility: **a)** in the process of heating and melting; **b)** in the process of draining the melt. 1. Dry box chamber; 2. Air evacuation hole; 3. Argon supply; 4. Quartz tube with a filter; 5. Vertical resistance furnaces; 6. Quartz tube with a crucible; 7. Alundum crucible for receiving the melt after the filter; 8. Alundum crucible with melt; 9. Branch pipe for pumping air; 10. Ceramic stand.

Based on the results of a series of preliminary experiments with metal meshes and ceramic porous filters, a cell-less mesh made of stainless steel P200 with linen weaving was chosen as the optimal type of phase separating partition (GOST 3187-76).

The structure and composition of the metal phases were studied using a JSM-5300 scanning electron microscope (SEM) (JEOL Ltd., Japan) with an ISIS energy-dispersive spectrometer (LinkAnalytical, Great Britain). The chemical analysis was carried out on a 725 ICP-OES inductively coupled plasma emission spectrometer (Agilent, USA/Australia).

Experimental techniques

At the first stage, the properties of the anode sludge simulator were studied. The composition of the real product is shown in Table 2; it is represented mainly by platinum group metals, zirconium, molybdenum and technetium. The presence of traces of alkali metals as well as uranium and chlorine is due to the partial capture of the salt electrolyte.

Table 2. Characteristics of the anode residue (semi-annual SNF conditioning)

Element	Content in sludge, wt. %	sludge activity, Bq/t	sludge energy release, kW/t
Tc	5.79	3.71E11	9.91E-1
Mo	22.68	4.25E-04	3.68E-20
Zr	18.56	4.50E15	6.16E-1
Ru	21.94	2.29E16	2.05E-1
Rh	6.60	2.27E-16	5.45E0
Ag	1.80	1.34E14	5.98E-2
Pd	17.30	1.27E10	1.89E-8
C	0.57	1.89E13	1.50E-4
K	1.20	–	–
Li	0.31	–	–
U	0.16	–	–
Cl	3.09	–	–
Total	100	5.03E16	6.33E0

To study the interaction of the anode sludge with cadmium, three cubes with a side of 7 mm and a weight of about 1.4 g each were cut out of metal ingots obtained in the electric arc furnace. One of them was used as a reference for visual assessment of external changes; the two others were kept in molten cadmium (200 g) under a layer of 3LiCl-2KCl (50 g) for 50 hours at a temperature of 500 °C. The resulting contact melt was submitted for chemical analysis to assess the migration of sludge components into cadmium. Part of the simulator was used to study the material microstructure.

At the next stage, experiments were carried out on the filtration of cadmium melts with metal powders simulating the components of the anode sludge. An alundum crucible containing 150 g of cadmium was placed in the furnace with a quartz tube of larger diameter with the addition of anode residue simulator components (5 g of each component in the form of powders of individual metals). The furnace was heated to a temperature of 490–500 °C. After

the cadmium was melted, it was kept under a layer of 3LiCl-2KCl (50 g) from two to four hours, depending on the distribution of simulators in the cadmium. Then the metal and salt were poured onto a filter preheated to 380–390 °C, which ensured the absence of crystallization of the melt and flux on the phase separating partition. The passage of the melt through the mesh was hampered by its viscosity and therefore the experiment was carried out with the creation of a rarefaction. However, part of the material remained on the filter in the form of an oxide film. At the end of the operation, the mesh was removed and weighed.

Discussions and results

In the study of the anode sludge simulators by the SEM method, it turned out that three zones are distinguished in the Alloy-1 sample, namely: upper, central and lower. Their photos in reflected electrons are shown in Fig. 2, and their composition is presented in Table 3.

Table 3. Zone compositions of the anode sludge simulator sample according to SEM and EDX data

Element	Content, wt. %				Average value
	Blend composition	Upper zone	Central zone	Lower zone	
Zr	19.8	23.83	14.55	17.87	19.19
Mo	23.7	15.52	32.83	20.80	24.75
Ru	23.7	21.32	28.25	19.63	24.79
Rh	6.7	8.79	4.51	5.54	6.65
Pd	18.2	28.08	11.44	18.48	19.76
Re	6.05	2.46	8.45	5.4	5.46

As can be seen from the table, all the three zones are close to the initial composition in terms of the average value. In the studied areas, there are two intermetallic phases: Phase-1 is dominant (light gray) concentrated in the form of an isometric form ranging in size from 20 to 150 µm.

The frame that ‘holds’ Phase-1 is Phase-2. The basis of the upper and lower zones is Phase-2, which forms rounded precipitates 5–15 µm in size. In the central part of the sample, both phases are developed in approximately equal amounts, just as in the upper and lower zones. Table 4 shows the composition of the phases for each zone.

Table 4. Phase compositions in different zones of the anode sludge simulator according to SEM and EDX data

Element	Content, wt. %					
	Upper zone		Central zone		Lower zone	
	Phase-1	Phase-2	Phase-1	Phase-2	Phase-1	Phase-2
Zr	1.43	37.51	0.90	36.90	1.80	36.15
Mo	51.75	1.4	50.67	2.48	51.16	1.56
Ru	27.74	24.47	24.49	28.77	29.68	27.36
Rh	0	11.27	0	12.34	0	12.11
Pd	1.69	25.11	1.13	19.32	1.38	22.24
Re	17.39	0	22.62	0	15.99	0

As can be seen, Phase-1 mainly consists of molybdenum; the shares of zirconium and palladium vary within 1–1.8 wt.%. Phase-2 is represented mainly by zirconium,

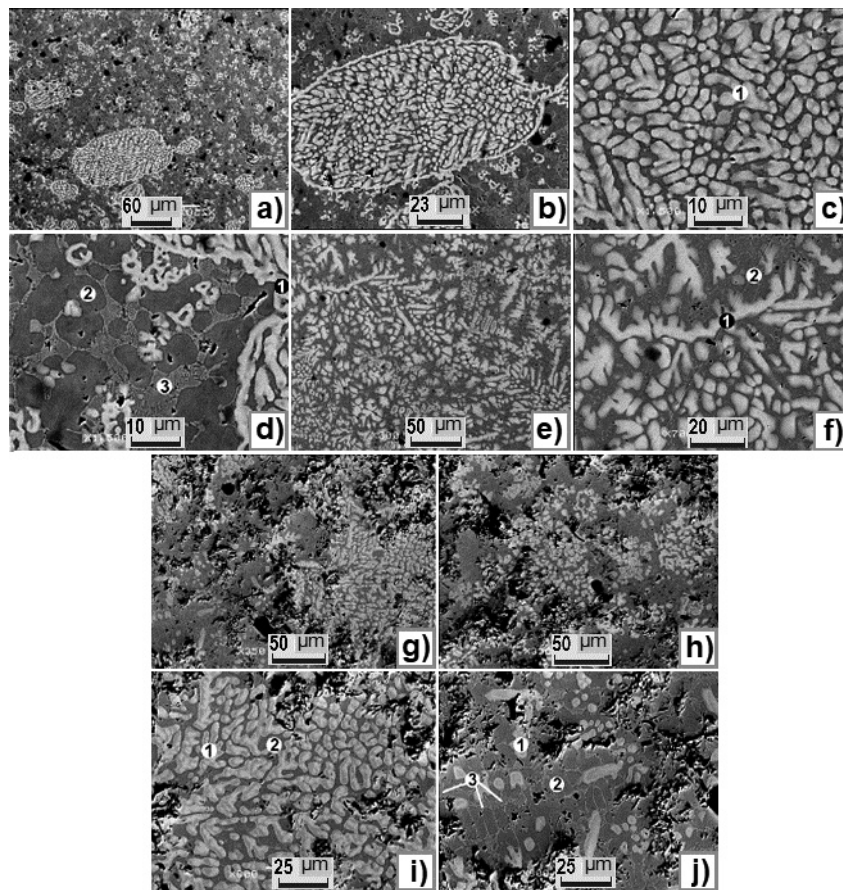


Figure 2. SEM images of the structure of the three zones of the Alloy-1 sample: a), b), c), d) Upper zone; e), f) Central zone; g), h), i), j) Lower zone. 1. Phase-1; 2. Phase-2; 3. Cryptogranular aggregate with a decay structure; the black inclusions are pores.

palladium and rhodium. Molybdenum is contained in small amounts (1–2.5 wt.%).

Element-by-element mapping of intermetallic inclusions in the central zone of the sample is shown in Fig. 3.

Fig. 4 shows a general view of the structure of the Alloy-2 sample and element-by-element mapping. Numbers 1–3 indicate the points of X-ray spectral microanalysis. As can be seen, molybdenum, rhenium, ruthenium, palladium and rhodium are uniformly distributed. Table 5 shows the chemical compositions of the identified phases.

Table 5. Composition of the intermetallic phases of the Alloy-2 sample according to the EDX data

Element	Blend composition	Content, wt.%			Average value
		Phase-1	Phase-2	Phase-3	
Mo	30.10	27.41	30.59	30.85	29.74
Ru	30.10	40.36	34.47	34.28	34.80
Rh	8.90	5.50	5.48	8.32	7.05
Pd	23.10	12.45	22.00	19.03	19.14
Re	7.70	14.28	7.46	7.53	9.24
Total	99.9	100	100	100.01	99.97

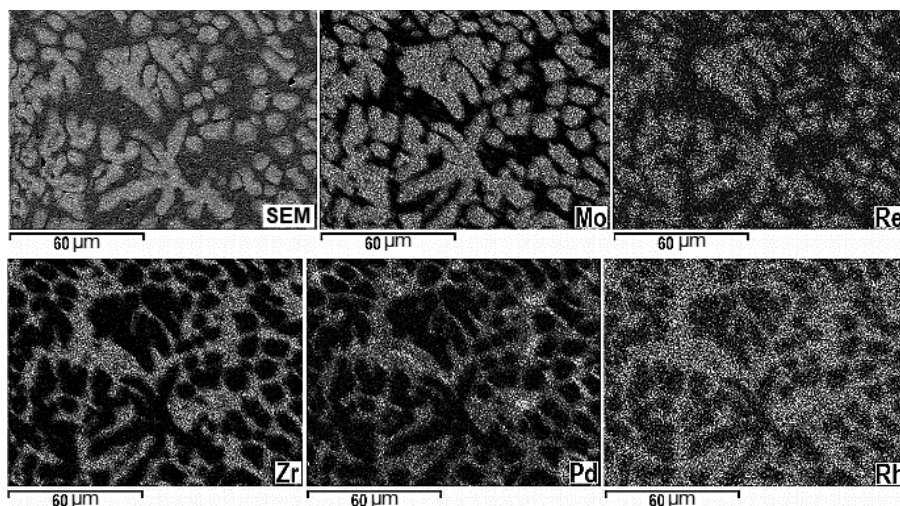


Figure 3. Distribution of elements in the central zone of the Alloy-1 sample over area in characteristic radiation.

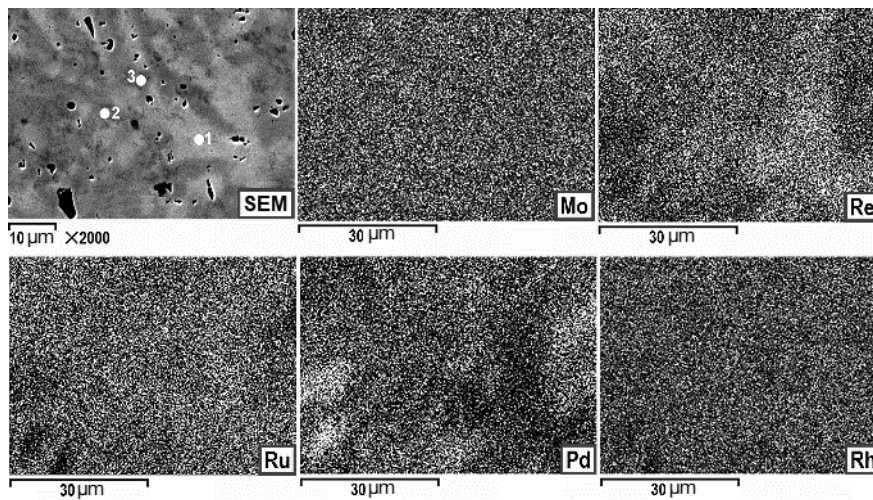


Figure 4. Distribution of elements of the Alloy-2 sample over area in characteristic radiation.

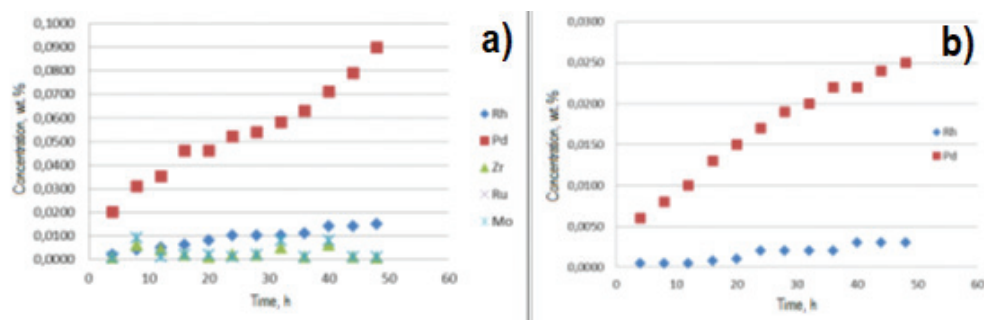


Figure 5. Change in the concentration of the metals in the cadmium melt: **a)** during the exposure of the alloy with zirconium (Alloy-1); **b)** during the exposure of the Alloy-2 sample.

From the data presented, it can be concluded that the compositions of Phases 2 and 3 do not differ fundamentally. The contents of palladium and rhodium from the ‘gray’ areas to the ‘dark gray’ ones varies from 22 to 19 wt.% and from 5.5 to 8.3 wt.%, respectively. The ‘lightest’ Phase-1 is predictably enriched in nuclei of the heavy element, rhenium (Reed 2005, Krishtal et al. 2009).

During the exposure of the samples in the cadmium melt under a flux, samples of the liquid metal phase were taken, based on the results of the analyses of which the leaching graphs presented in Fig. 5 were constructed.

From the Alloy-1 sample (see Fig. 5a), mainly palladium and rhodium pass into the melt (up to 0.07 and 0.015 wt.%, respectively); zirconium and molybdenum are also found in cadmium, but in trace amounts. In the Alloy-2 sample, the cadmium melt was enriched only with palladium and rhodium, which indicates a greater stability of the product in comparison with the sludge containing zirconium. The appearance of the samples with a side of 7 mm, kept in the cadmium melt at 500 °C for 50 hours, is shown in Fig. 6.

It can be seen that the original color of the Alloy-2 sample has changed to ‘black’, but the facets and edges have retained their outlines. On the cube of Alloy-1 containing zirconium, cavities are observed, the edges are rounded, and the surface is covered with a black powdery coating. According to the results of the analysis, it was

found that the dispersed material is Phase-1 and intermetallic $ZrCd_3$, which indirectly indicates the interaction of Phase-2 with cadmium, accompanied by the migration of palladium and rhodium into the melt.

It can be stated that in the case of the formation of an anode deposit in the form of a collective alloy, filtration purification of liquid media of SNF electrorefining can remove only a part of FPs that do not form true solutions with cadmium. Palladium and, to a lesser extent, rhodium will pass into the anode and gradually accumulate in it, which will subsequently require a fundamentally different approach to purifying the liquid metal medium. For this purpose, vacuum distillation or rectification can be proposed, which is widely used for the purification of metals characterized by a low boiling point (Kato et al. 2005, Volodin et al. 2013, Koval 2016).

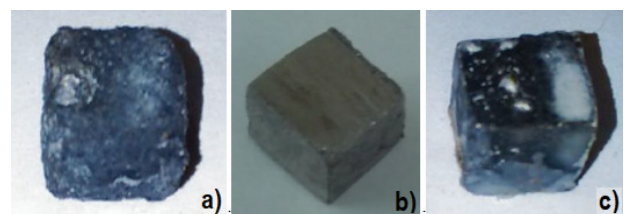


Figure 6. Appearance of the samples with a side of 7 mm before and after the exposure: **a)** the original sample; **b)** the Alloy-1 sample after the exposure; **c)** the Alloy-2 sample after the exposure.

In the process of filtering the Cd-Mo system, molten cadmium and salt passed through the filter, the molybdenum separation efficiency was 99%, and the remaining 50 mg of metal powder were distributed in the electrolyte. Fig. 7 shows the appearance of the filter and a two-phase ingot 20 mm high after the experiment.

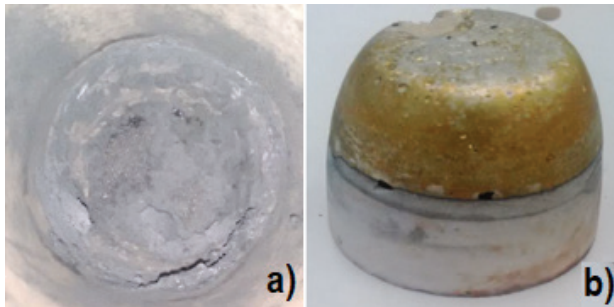


Figure 7. Appearance of the P200 filter mesh and ingot after the experiment: **a)** P200 filter; **b)** ingot 20 mm high after passing the P200 filter.

In the case of the Cd-Mo-Re-Ru-Pd system, cadmium and electrolyte melts were also filtered out without hindrance. The mass of the purified metal was 127.9 g, and the sediment on the filter was 24.2 g. The efficiencies of trapping palladium and ruthenium on the partition did not exceed 13.1 and 54.3%, respectively, which is associated with the thinning of the powders due to their partial dissolution in cadmium. Molybdenum and rhenium, which

obviously did not interact with the melt, separated almost completely in an amount of about 99.3% each.

Conclusions

As a result of the work, it was found that in the case of the formation of FPs of a collective alloy in the process of electrorefining, the purification of anode cadmium by filtration will make it possible to remove only a part of FPs. Zirconium, which forms an intermetallic composition $ZrCd_3$, molybdenum, and technetium, which are not wetted by cadmium, can be removed from the melt using a phase separating partition based on a P200 type steel filter mesh. The recovery of Mo and Re is more than 99% when using a P200 type mesh. In the case of joint filtration of the metal and salt phases, the breakthrough is assimilated by the electrolyte, particles are not enlarged, but they can be collected into agglomerates.

The achieved filtration efficiency of Pd and Ru powders did not exceed 13.1 and 54.3 wt.%, which was due to their thinning due to partial dissolution. Palladium and, to a lesser extent, rhodium passing into the melt will accumulate in cadmium over time, increasing heat generation, which will require the use of a fundamentally different refining method, such as vacuum distillation. It is shown that the metal melt in contact with the collective alloy of FPs is saturated with Rh and Pd to 0.01 and 0.025%, respectively, in 50 hours at 500 °C.

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