

RECEIVING ELECTROLYTIC NICKEL-CONTAINING POWDERS OF THE PREDICTED STRUCTURE AT ANODE DISSOLUTION OF SECONDARY HEAT RESISTING ALLOYS

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Introduction

The secondary heat resisting nickel alloys containing rare metals and, in particular, rhenium processed by pyro-, hydro- and electrometallurgical ways now. Among the pyrometallurgical the methods connected with oxidation and chlorination, hydrometallurgical – with acid leaching and the subsequent sorption of rare metals are most known, electrometallurgical are based on anode dissolution, electrolysis and electro dialysis. But most often use after all combined methods when for complex processing carry out all above-stated methods. Processing of heat resisting alloys is complicated by the high content of a number of non-ferrous metals and mutual influence of components of alloys on technological processes [1-6]. At the same time in literature problems of receiving powders of non-ferrous metals which is possible when processing such alloys are a little consecrated. Therefore the purpose of the work is to show a possibility of receiving electrolytic nickel-containing powders of the predicted structure at anode dissolution of secondary heat resisting alloys.

Experimental

As an object of researches heat resisting alloy of the following structure has been chosen (masses. %): Re - 4,0; Co - 9,3; W - 8,6; Y - 0,005; La - 0,005; Al - 6,0; Cr - 5,0; Ta - 4,0; Nb - 1,6; Mo - 1,1; With - 0,16; B - 0,15; Ce - 0,025, Ni-60,05. Alloy represents difficult system at which along with non-ferrous metals there are rare and rare-earth elements.

For processing of the specified secondary nickel-containing alloy with receiving powders of non-ferrous metals used a method of anode dissolution with the subsequent sedimentation of nickel and the accompanying metals on the titanic cathode.

Anode dissolution of secondary heat resisting nickel-containing alloy was performed in the galvanostatic mode using acid

electrolytes – muriatic, sulfate and nitrate. Preliminary experiments showed that most effectively dissolution proceeds with use of electrolytes with concentration of acids of 100 g/l.

Content of non-ferrous and rare metals as a part of electrolyte was controlled by a photometric method, besides, made the element analysis with use of ICP of a mass spectrometer for the isotope and element analysis of ELAN DRC-e (Perkin Elmer, Canada).

The phase composition of the received powders was carried out with use of the Shimadzu XDR 6000 diffractometer (radiation CuK α , rotation of a sample, continuous (1 hail/min.), step-by-step (a step 0,02 °, an exposition 10s) by the modes in the range of corners 2 θ 5-80 °).

Determination of the sizes of particles by method of measurement of dynamic light scattering is executed on the analyzer of the size of submicronic particles and dzeta-potential of Delsa™ Nano, PN A54412AA.

The polarization phenomena in the course of anode dissolution is studied on the electrochemical technological complex (development of LLC IP Tetran, the Russian Federation) using not compensation way of measurement of potential.

It is known that the polarizing agent promotes formation of a set of the centers of crystallization and by that to increase in dispersion of the received electrolytic powders of metals.

In the real work the polarizing effect of the following organic acids is considered: sulfosalicylic, sulfamic and malic.

Results

At anode dissolution of heat resisting alloy in nitrate electrolyte influence of current on a bathtub which was varied within I = 0,5-2,5A, on compositions of the received electrolyte (within 3 hours) (table 1) and anode slime (table 2) was estimated.

Table 1. Influence of current on composition of nitrate electrolyte at anode dissolution of nickel-containing heat resisting alloy

I, A	Content of metals in electrolyte, g/l					Dissolution rate, g/h*cm ²	Current efficiency for nickel, %
	Ni	Re	Co	Al	Cr		
0,5	10,9	1,06	0,46	0,08	0,05	0,030	58,5
1,0	10,43	1,08	0,35	0,10	0,05	0,045	59,1
1,5	12,1	1,13	0,39	0,18	0,11	0,055	58,9
2,0	15,65	1,37	0,56	0,21	0,18	0,056	49,9
2,5	17,03	1,86	0,61	0,29	0,20	0,055	41,3

Table 2. Chemical composition of anode slimes

I, A	Content of metals, %								
	Al	Cr	Co	Ni	Re	Nb	Mo	Ta	W
0,5	7,63	3,88	6,23	41,09	2,83	3,18	3,89	11,43	19,84
1,0	6,98	3,95	5,23	25,98	1,93	5,58	6,22	15,35	28,78
2,5	5,23	1,88	4,23	27,49	1,83	6,18	7,89	15,43	29,84

The structure of a cathodic deposit was influenced not so much by the electrolysis mode, how many the nature of the used acid electrolyte. Results of researches are shown in table 3.

Table 3. Influence of the nature of electrolyte on structure of cathodic deposites

Electrolyte	Duration, h	Content of metals, %			
		Ni	Co	Re	Al
100 g/l H ₂ SO ₄	1	94	5	-	trace
	2	95	4	-	trace
	3	95	4	-	trace
	4	96	3	-	trace
100 g/l HNO ₃	1	54	17	16	13
	2	52	18	16	14
	3	54	16	16	14
	4	55	14	15	16
100 g/l HCl	1	68	16	-	16
	2	66	18	-	16
	3	66	18	-	16
	4	64	19	-	17

The data on studying of influence of the polarizing agents obtained during the researches – sulfosalicylic, sulfamic and malic acids have shown that their presence doesn't affect structure of a cathodic deposit, but exerts impact on particle size distribution of the received powder. Results of researches of particle size distribution of the received powders have shown that the greatest impact is exerted by introduction of sulfosalicylic acid thanks to which the share of small fraction (less than 0,1 microns) increased to 99,5-99,7% (the figure 1 – on the example of nitrate electrolyte).

Discussion

Apparently from table 1, the maximum exit of nickel on current is observed at current 1,0 A. The nature of transition of rhenium, cobalt, aluminum and chrome to composition of electrolyte at electrolysis is similar to behavior of nickel.

On the basis of the data presented in table 2 follows that at increase in current at anode dissolution, anode slime is impoverished by nickel, cobalt, chrome and rhenium, but it is considerably enriched with rare metals – niobium, tantalum, molybdenum and tungsten and actually represents their concentrate that can be processed by methods of ammoniac hydrometallurgy.

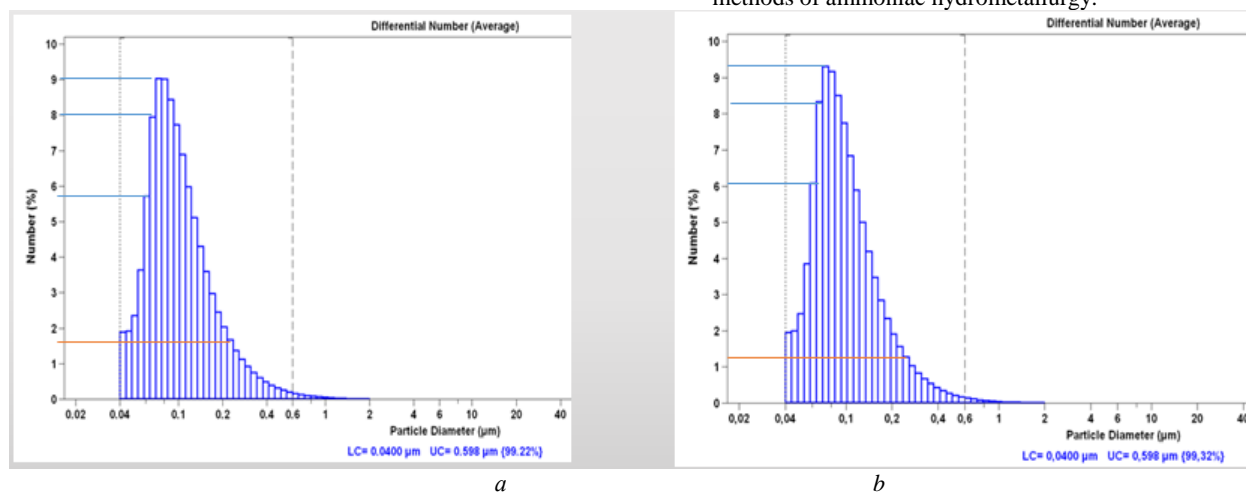


Figure 1. Particle size distribution of the cathodic deposit received at anode dissolution of heat resisting nickel alloy from nitrate electrolyte (a) and nitrate electrolyte with additive of 10 g/l of sulfosalicylic acid (b)

The chemical composition of the received cathodic deposit is established and the link of structure with the used electrolyte is revealed. It is shown (table 3) that depending on the nature of electrolyte and conditions of carrying out electrochemical process – parameters of process and the current program – cathodic deposits of various quality can be received:

- from sulfate electrolyte – nickel-containing powder with the content of nickel about 95%;
- from nitrate electrolyte – nickel-containing powder of structures: Ni:Re:Co:Al = 3:1:1:1 and Ni:Re:Co:Al = 10:1:1:1;
- from muriatic electrolyte – nickel-containing powder of structure Ni:Co:Al = 4:1:1.

On the basis of the results presented in the figure 1 follows that among the organic acids used as the polarizing agents on dispersion of the received powder the greatest impact is exerted by sulfosalicylic acid.

Thus, the possibility of receiving nickel-containing powder of the predicted structure and dispersion at electrochemical processing of secondary heat resisting alloy with use of acid electrolytes is considered. It is shown that the structure and dispersion of the received powder are influenced by the choice of the nature of acid, the mode of anode dissolution and addition to electrolyte of the polarizing agent.

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