Electrochemical Solution of Molybdenum Mixture

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ABSTRACT

When molybdenum is processed by powder metallurgy, insoluble molybdenum compounds arise, which form specs on the surface of nickel boats (Ni - 63%, Mo - 27% alloy). It is necessary to search for an effective technology for cleaning nickel boats from molybdenum cake, ensuring their repeated use and return to the production cycle of metal bound in the cake [1, 2, 3, 4, 5].

To solve this problem, we applied electrochemical methods, i.e. conditions for the electrochemical dissolution of molybdenum compounds (metallic molybdenum, nickel-molybdenum alloy, molybdenum cake and nickel-molybdenum boat with cake) in an aqueous electrolyte of molybdenum acid ammonium, additionally containing caustic potassium [6, 7].

An electrochemical technology has been developed for cleaning Ni-Mo boats from molybdenum cake, which ensures their repeated use and the receipt of an additional amount of metal from the solution. The calculations show a sufficiently high selectivity of dissolution of molybdenum and high economic efficiency of the proposed technology.

Keywords: molibden, electrochemical solution, molibden mixture, Ni-Mo alloy, current density, selective dissolution.

Purpose (Object): To study the selective composition and conditions of electrochemical dissolution of molybdenum compounds

Introduction: In metallurgical industries there are waste cakes, sludge tailings and scrap containing non-ferrous and precious metals. They are technogenic raw materials for the extraction of non-ferrous and precious metals. Involvement in the processing of such metal-containing secondary raw materials (copper clinker of zinc production, lead scrap in battery production, scrap from jewelry refineries and alloys for dentures, special solders, etc.) is a potential source of additional capacity building for the production of non-ferrous and precious (noble) metals in the metallurgy of the Republic of Uzbekistan [1, 2, 3, 4, 5].

The development of technology for electrochemical refining of such non-ferrous and other valuable metals from secondary raw materials is an urgent problem [6, 7]. For the separation and extraction of non-ferrous metals, preference is given to the hydrometallurgical method, in particular electrochemical refining.

The dissolution of copper clinker containing copper, zinc, cadmium, lead, iron and noble metals has been studied. The dissolution and isolation of non-ferrous metals was carried out from dry sulphated raw materials by leaching a 10-15% solution of sulfuric acid. As a result, all non-ferrous metals go into solution and precious metals are concentrated in the leaching cake [8]. Non-ferrous metals were extracted from the solution by known methods, and precious metals were extracted from the cake.

During the reduction of ammonium steam molybdate in nickel boats (Ni alloy - 63%, Mo - 27%) in a hydrogen environment, part of the metal sinters and sticks to the inner surface of the boats.

During the processing of molybdenum by powder metallurgy, insoluble molybdenum compounds appear, which form cakes on the surface of nickel boats (Ni alloy - 63%, Mo - 27%), strongly associated with it and cannot be removed, due to which nickel boats cannot be reused. used and become a waste of production and so far there is no way to process such secondary metals.

It is necessary to search for an effective technology for cleaning nickel boats from molybdenum sinter, which ensures their repeated use and return to the production cycle of metal bound in sinter.

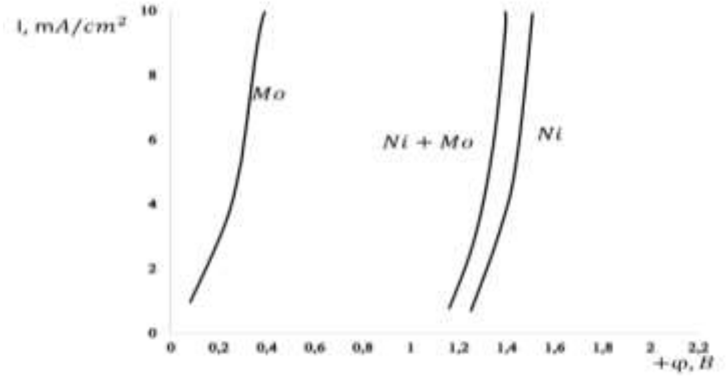
The purpose of this work is to develop an electrochemical method for dissolving molybdenum compounds from nickel-molybdenum sinter. To solve this problem, electrochemical methods were used, i.e. anodic dissolution of molybdenum compounds in an aqueous electrolyte of ammonium molybdenum acid, additionally containing caustic potassium.

From the literature data, it is known that there is no selective solvent during the electrochemical anodic dissolution of a molybdenum compound from the surface of a nickel–molybdenum alloy [1–5].

Objects and methods of research. For electrochemical research, molybdenum-containing raw materials were presented: metallic molybdenum, nickel-molybdenum alloy, molybdenum cake and nickel - molybdenum boat with cake.

To dissolve the above samples, after many searches for a solvent (potassium nitrate, alkaline solution of oxalic acid, caustic potash, etc.), a salt electrolyte of ammonium molybdate (NH4)6Mo7O24*4H2O was selected. However, molybdenum, molybdenum speck, and the nickel boat itself dissolve in this solution. The method of polarization measurement was used to find the selective solvent and the dissolution potential of the components. The experiments were carried out on small electrodes, prepared respectively from a molybdenum, nickel plate and a nickel-molybdenum boat body at a temperature of 30 0C. The polarizing current was supplied from a P-5827 potentiostat. The potential was measured with an A4-MG cathode voltmeter [2, 8].

Results and its discussion. An analysis of the measurements performed showed that it is necessary to add a passivating component to the electrolyte, which does not prevent the dissolution of the molybdenum cake, but passivates the surface of the boat. Such a component turned out to be sodium or potassium alkali. With the introduction of 50-100 g/l of alkali into the salt electrolyte, a sharp change is observed in the mutual arrangement of the polarization curves over the potential range (Fig.). On this basis, for the cleaning of nickel boats, we propose the following composition of the electrolyte (g/l): molybdic acid ammonium - 20-30, caustic potash - 50-100. In such an electrolyte, metallic molybdenum remains in the potential range when passivation of its surface is not achieved, and the Ni-Mo alloy and metallic nickel are passivated, i.e. molybdenum cake dissolves, but the boat itself does not dissolve.



Rice. Fig. 1. Anode polarization curves of dissolution of molybdenum, nickel and molybdenum-nickel alloy in alkaline electrolyte. Temperature 30 0C.

The electrolysis regime was experimentally established. The process of anodic dissolution is carried out at a current density of 80 - 120 A/dm2, voltage of 5 - 6 V, electrolyte temperature of 35 - 55 0C, pH - 9-10. This electrolyte composition and electrolysis conditions were used for anodic dissolution of molybdenum, as well as other compounds and alloys, such as molybdenum, molybdenum-rhenium, molybdenum sinter-nickel-molybdenum boat with sinter and nickel-molybdenum alloy. Data on the electrochemical dissolution of these compounds are given in the table.

From the table it follows that in the alkaline electrolyte of molybdenum the molybdenum cake dissolves, and the boat itself remains intact. To carry out experiments on cleaning nickel-molybdenum boats with sinter, a special bath was designed. The current output is 55-65%.

Table

Rate of anodic dissolution of molybdenum compounds.

Current density 60 A / m2, temperature - 25 0C, time - 2 hours

№	molybdenum compound	Solvent composition			
		$(NH_4)_6Mo_7O_{24}*4H_2O$		$(NH_4)_6Mo_7O_{24}*KOH$	
		Dissolved, g	Dissolution rate, mg/cm2 hour	Dissolved, g	Dissolution rate, mg/cm2 hour
	Molybdenum Mo	0,155	12,88	0,833	69,4
	Mo-Re	0,4372	36,40	0,978	81,53
	NiMo	0,206	17,15	0,744	62,03
	boat with speck	0,139	11,52	0,821	69,23
	Speck Mo	0,0064	0,53	0,0002	0,018

After establishing the composition of the electrolyte and the electrolysis mode, we proceeded to clean the nickel boats from molybdenum cake. The sinter surface in a small boat was equal to 1 dm2, and the amount of sinter did not exceed 400 g. At a current density of 100 A/dm2, the minimum cleaning time was 6.7 hours, and at a current density of 30 A/dm2, respectively, a day.

For the experiments, a special design of the electrolyzer and the cathode as a whole made of stainless steel was developed, equipped with a cooling device, perforated with a steel stand for boats (anodes), which were connected to the anode terminal of the VAKR-3200/24 rectifier and a plate cathode with a vertical conductor rod. A perforated polyvinyl plate was installed between the sinter surface (anode) and the cathode to protect against short circuits. All boats are placed on an anode grid with an intermediate distance of 1.5-2 cm, which is connected by a steel conductor. The electrolyte volume is 170 l. The height of the grate above the bottom of the bath is about 75 mm with a straightener. The resulting sludge accumulates on the inclined bottom of the bath and is removed by accumulation. It has been established that in the process of anodic dissolution of molybdenum in the electrolyte, the content of molybdenum rises to 300-400 g/l in terms of metal. The alkali content in the course of electrolysis must be corrected and maintained at the level of 50-100 g/l. The consumption of alkali per 1 kg of molybdenum is 1.17-1.80 kg due to the formation of an electrolyte above the mirror. Under these electrolysis conditions, assuming that each bath contains 400 g of molybdenum cake, saturated solutions of 350 g/l can be obtained in three days. The molybdenum-containing solutions obtained in the process of anodic dissolution of the sinter are sent to the hydrometallurgical process for the extraction of molybdenum salts.

Conclusions.

An electrochemical technology has been developed for cleaning nickel boats from molybdenum sinter, which ensures their repeated use and return to the production cycle of metal bound in sinter. The calculations carried out show a rather high selectivity of molybdenum dissolution and high economic efficiency of the proposed technology.

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