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RESEARCH ARTICLE

Application of pulse current for dissolution of heat-resistant GS32-VI alloy

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Abstract

Objectives. *To identify the regularities of electrochemical processing of the heat-resistant GS32-VI alloy in a sulfuric acid electrolyte with a concentration of 100 g/dm^3 under the action of a pulsed current in a pulsed mode.*
Methods. *Using the electrochemical technological complex EHK-1012 (developed by IP Tetran) and a non-compensatory method of measuring potential, polarization and depolarization curves with a change in pulse duration and a pause between them were recorded. The current pulses had an amplitude ranging from 0 to 3.5 A (when recording the polarization and depolarization curves), pulse durations ranging from 200 to 1200 ms, and a pause (delay) between pulses ranging from 50 to 500 ms. There were no reverse current pulses.*
Results. *The parameters of the current program that provide the maximum values of the alloy dissolution rate and current output were determined: with a current pulse amplitude of 2 A, a current pulse duration of 500 ms, and a pause duration between pulses of 250 ms, the maximum dissolution rate of the alloy is 0.048 g/h·cm^2, while the current output for nickel is 61.6% with an anode area of 10 cm^2. The basic technological scheme for processing the heat-resistant GS32-VI alloy, which includes anodic alloy dissolution in a pulsed mode, is proposed.*
Conclusions. *Electrochemical dissolution of GS32-VI alloy under pulsed current action results in an optimal dissolution rate ratio of the alloy components, ensuring the production of a cathode precipitate with a total nickel and cobalt content of 97.5%.*

Keywords: *pulse current, sulfuric acid, GS32-VI alloy, polarization and depolarization curves, amplitude of pulse current, duration of pulse current, pause between pulse current*

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НАУЧНАЯ СТАТЬЯ

Применение импульсного тока для растворения жаропрочного сплава ЖС32-ВИ

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Аннотация

Цели. *Выявить закономерности электрохимической переработки жаропрочного сплава ЖС32-ВИ, проводимой в импульсном режиме в сернокислом электролите с концентрацией 100 г/дм^3 под действием импульсного тока.*
Методы. *Снятие поляризационных и деполяризационных кривых с изменением длительности импульса и паузы между ними проводили с помощью электрохимического технологического комплекса ЭХК-1012 (разработан ООО ИП «Тетран»), использующего некомпенсационный способ измерения потенциала. Амплитуда импульсов тока находилась в диапазоне значений от 0 до 3.5 А (при снятии поляризационных и деполяризационных кривых), длительности импульсов изменялись от 200 до 1200 мс, пауза (задержка) между импульсами – от 50 до 500 мс, импульсы реверсивного тока отсутствовали.*
Результаты. *Определены параметры токовой программы, обеспечивающие максимальные значения скорости растворения сплава и выхода по току. При амплитуде импульса тока 2 А, длительности импульса тока 500 мс и продолжительности паузы между импульсами 250 мс максимальная скорость растворения сплава 0.048 г/ч·см^2, при этом выход по току для никеля равен 61.6% при площади анода 10 см^2. Предложена принципиальная технологическая схема переработки жаропрочного сплава ЖС32-ВИ, включающая анодное растворение сплава в импульсном режиме.*
Выводы. *Электрохимическое растворение сплава ЖС32-ВИ под действием импульсного тока способствует оптимальному соотношению скоростей растворения составляющих сплава, что обеспечивает получение катодного осадка с суммарным содержанием никеля и кобальта 97.5%.*

Ключевые слова: *импульсный ток, серная кислота, сплав ЖС32-ВИ, поляризационная и деполяризационная кривые, амплитуда импульса тока, длительность импульса тока, пауза между импульсами тока*

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INTRODUCTION

The high cost of nickel heat-resistant alloy components (rhenium, tantalum, cobalt, etc.) that have reached the end of their service life necessitates their return to industrial production. Processes based on electrochemical methods, which allow for the regeneration of rare refractory metal wastes with high technological and economic indicators, are the most effective and promising paths in metal waste processing technology [1–15]. The literature describes various solutions to the problem under discussion. Thus, processing methods based on opening processed raw materials with acid solutions are well known. The use of sulfuric, hydrochloric, and nitric acids followed by the electrochemical release of the target products at a fixed current density [1–5] is the most common. Another approach that involves the anodic dissolution of the processed metal-containing raw material and subsequent separation of commercial products from the electrolyte solution is described in [6–17].

The dissolution and transfer rate of products into solution during the anodic dissolution of metals or alloys is significantly influenced by the current program. It is worth noting that anodic dissolution processes, in which the magnitude and direction of the current used are variable, are common in the processing of various types of secondary raw materials. Such modes, in contrast to electrochemical processes with a constant current value, are called unsteady electrolysis modes or unsteady electrolysis. In papers [6–11, 13], the dissolution process is carried out using alternating currents of various kinds. The implementation of the anodic dissolution process under these conditions prevents passivation phenomena and increases the anodic dissolution rate.

Unsteady electrolysis in pulse mode has been effectively used in the production of coatings and the electrochemical treatment of materials [17–27]. However, despite the superior characteristics of electrochemical processes, its use in the processing of secondary raw materials is limited.

This work aims at revealing the regularities of the electrochemical processing of the heat-resistant GS32-VI alloy in a sulfuric acid electrolyte in the pulse mode.

EXPERIMENTAL

Electrochemical dissolution of the alloy composition (wt %) 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; C–0.16; B–0.15; Ce–0.025, Ni–60. The polarization and depolarization curves were obtained using electrochemical technological complex EHK-1012 (developed by *IP Tetran*), which uses non-compensation method of potential measurement [28]. The reference electrode was a silver chloride electrode.

Current diagram for polarization and depolarization curves: current pulse amplitude varied from 0 to 3.5 A, pulse duration varied from 200 to 1200 ms, pause pulse (delay) varied from 50 to 500 ms, and no reverse current pulses were used (Fig. 1).

The electrochemical cell consisted of a 300 mL fluoroplastic reservoir, a fragment of a blade from the heat-resistant GS32-VI alloy with an area $S = 10$ cm^2 (anode), and a titanium (VT1-0) plate with an area $S = 9.5$ cm^2 (cathode).

Electrode weight loss was determined on analytical scales ANDGR-300 with an accuracy class of +/−0.0001 g. The process was conducted at a temperature of 20–25°C. Elemental analysis was performed using an ICP mass spectrometer for isotopic and elemental analysis ELAN DRC-e (*PerkinElmer*, Canada).

RESULTS AND DISCUSSION

Based on literature data and previously conducted studies, the feasibility of using acidic electrolytes based on sulfuric and nitric acids for processing of nickel-based refractory alloys was revealed [1, 4, 5, 9, 10, 13, 14]. We selected sulfuric acid at a concentration of 100 g/dm^3, which provides a high dissolution rate, to obtain a cathodic nickel-containing product. Lower acid concentrations result in a lower alloy dissolution rate (less than 0.030 g/h·cm^2), while sulfuric acid concentrations of 250 g/dm^3 and higher enhance the probability of increased anode sludge.

The main advantage of pulsed electrolysis is the ability to make abrupt changes in the electrode potential, which significantly affects the charge transfer stage, adsorption and crystallization, the overall kinetics of the process, and determines the course of each electrochemical reaction.

When using pulse current, the significant parameters are pulse duration and amplitude, pause between pulses, and presence of reversing pulse. To analyze the effect of these parameters on the process of electrochemical dissolution of the heat-resistant GS32-VI alloy, anodic polarization curves with changes in the pulse duration and the pause between them were taken. Depolarization curves were taken after the current pulse to show the dynamics of the electrode potential.

Pulsed current, unlike direct current, allows for effective control of electrode processes. The steep leading edge of the pulses allows for a faster increase in electrode potential from the minimum to the maximum value (Fig. 1). As a result, electrolyte depletion near the electrode is accelerated, current lines are redistributed, and fine crystalline precipitates are formed on the cathode, resulting in the production of smaller anode sludge particles in the case of soluble anodes with heterophase composition. This is presumably true in anodic dissolution since GS32-VI is a multicomponent, heterophase alloy. The main elements of its phase composition are dispersed (<0.5 µm) 1-phase particles γ, which are based on the ordered intermetallic compound Ni_3Al (superstructure L12) and a complexly alloyed nickel γ-solid solution, as well as carbide and boride phases of various types [29]. Simultaneously, the steep decline of direct pulses causes an increase in the potential swing, which affects the precipitation structure as well as some electrolysis process parameters.

Figures 2 and 3 show polarization and depolarization curves obtained in pulsed galvanodynamic mode, respectively.

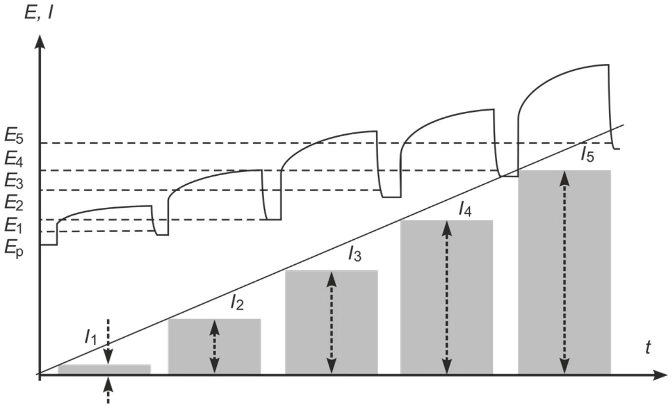

Fig. 1. Current diagram for polarization and depolarization curves.

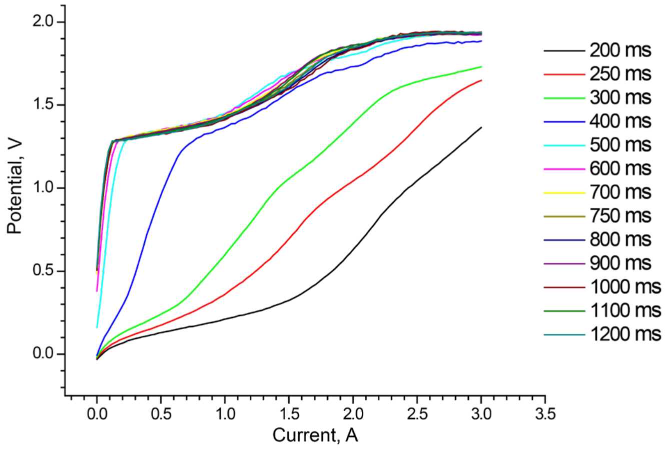

Fig. 2. Polarization curves of GS32-VI alloy dissolution in a sulfuric acid electrolyte with a concentration of 100 g/dm^3 at different pulse durations and a pause of 250 ms.

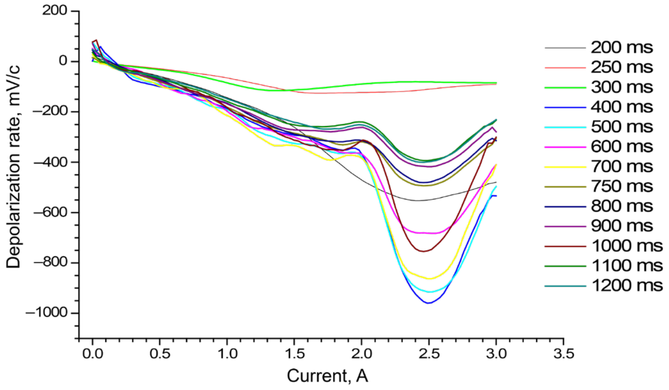

Fig. 3. Depolarization curves of GS32-VI alloy dissolution in a sulfuric acid electrolyte with a concentration of 100 g/dm^3 at different pulse durations and a pause of 250 ms.

Figure 2 illustrates that with a polarization time of 200–300 ms, the shape of the polarization curve has a smoothed form from the obtained dependences of the shape of polarization curves on the pulse duration. When the current pulse duration is increased up to 500 ms, a horizontal area corresponding to the maximum dissolution rate of the alloy appears on the polarization curve. Further increase in pulse duration (up to 1200 ms) has no noticeable effect on the shape of polarization curves.

The described dependences on the influence of current pulse duration on the shape of polarization curves are confirmed by the obtained depolarization curve dependences. At current pulse durations of 400–500 ms, the depolarization curves clearly show areas corresponding to different electrochemical processes (Fig. 3).

The polarization and depolarization curves of GS-32VI alloy dissolution in sulfuric acid electrolyte with a concentration of 100 g/dm^3, a pulse duration of 500 ms, and a delay of 250 ms are shown in Fig. 4 to highlight the areas corresponding to the course of various electrochemical processes.

I area: E = 0.18–1.78 V—the dissolution of the complex-alloyed nickel γ-solid solution phase occurs;

II area: E = 1.78–1.91 V—dissolution γ of 1-phase based on the ordered intermetallic compound Ni_3Al;

III area: $E \geq 1.91$ V—release of oxygen through the formation of intermediate unstable oxides.

Using similar dependencies, the effect of the pause duration between pulses on the shape of polarization and depolarization curves was determined (Fig. 5, 6). The pause in pulsed electrolysis had a significant effect on the metal deposition process and the quality of the resulting cathode precipitation. As a result of migration, the concentration of ions in the near-electrode space equalizes completely or partially depending on the pause duration. Passivation of the electrode surface is also possible, which involves the oxidation or adsorption of impurities in the electrolyte solution on the active parts of the electrode surface.

During GS-32VI alloy dissolution in a sulfuric acid electrolyte with a concentration of 100 g/dm^3 using pulsed current, a high alloy dissolution rate was attained at a pulse duration of 500–700 ms and a pause pulse delay of 250 ms. The dissolution rate ratio of nickel, cobalt, and rhenium provides a cathode deposit with a nickel and cobalt content of at least 90%

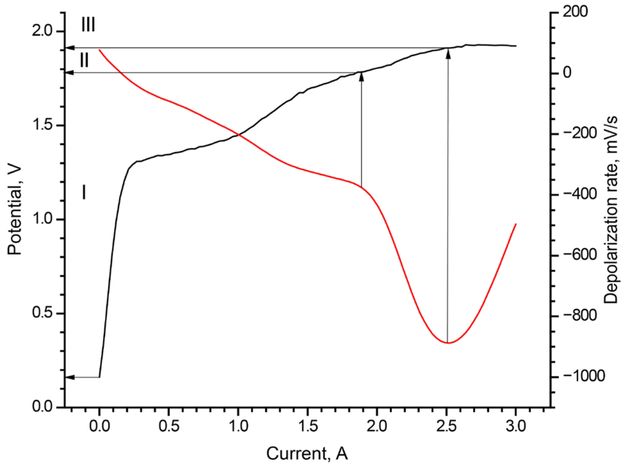

Fig. 4. Polarization and depolarization curves of GS32-VI alloy dissolution in the sulfuric acid electrolyte at a concentration of 100 g/dm^3, 500 ms pulse duration, and a delay of 250 ms.

The maximum current pulse amplitude during anodic alloy dissolution is limited to 2.5 A (at anode area S = 10 cm^2); exceeding this value results in an undesirable process–oxygen evolution, which reduces current yield. The optimal value of the current pulse amplitude is determined based on the maximum alloy dissolution rate combined with a high current yield of nickel current yield, which is the base of GS32-VI alloy.

Table 1 shows experimental data results on the effect of current pulse amplitude on the anodic dissolution rate of GS32-VI alloy in sulfuric acid electrolyte with a concentration of 100 g/dm^3. For each experiment, a new anode, which was a heat-resistant GS32-VI alloy blade fragment with an area S = 10 cm^2.

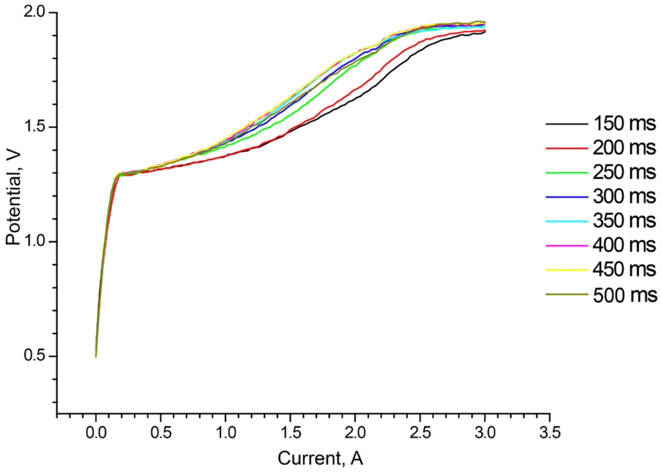

Fig. 5. Polarization curves of GS32-VI alloy dissolution in a sulfuric acid electrolyte with a concentration of 100 g/dm^3, a pulse duration of 500 ms, and different pause times.

The dissolution rate of the alloy v was calculated using the mass loss of the dissolved alloy according to the formula:

$$v = (m_1 - m_2)/S{\cdot}t^{-1},$$

where m_1 is the initial mass of the dissolved sample, m_2 is the mass of the sample after electrochemical dissolution, S is the working area of the electrode, and t is the processing time.

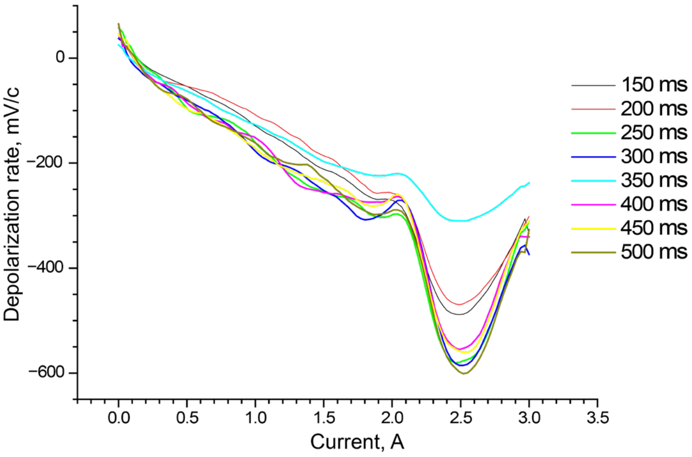

Fig. 6. Depolarization curves of GS32-VI alloy dissolution in a sulfuric acid electrolyte with a concentration of 100 g/dm^3, a pulse duration of 500 ms, and different pause times.

Based on the data obtained, the following technological parameters for reprocessing the GS32-VI alloy by the electrochemical method were selected: current pulse amplitude—2 A (at the anode area S = 10 cm^2), current pulse duration—500 ms, and the pause between pulses—250 ms.

In the pulsed galvanostatic mode, a total of 97.56 g of heat-resistant GS32-VI alloy was processed in a sulfuric acid electrolyte with a concentration of 100 g/dm^3. Afterward, 55.08 g of cathode deposit and 27.32 g of anode sludge were received; their chemical composition is summarized in Table 2.

Thus, refractory metals, such as niobium, tantalum, molybdenum, and tungsten are concentrated in the anode sludge, whereas cobalt and rhenium, as well as the majority of aluminum, chromium, and nickel, pass into the electrolyte in small amounts. It is worth noting that electrochemical processing with pulsed current allows for rhenium concentration in the anode sludge, with a content of 77% of the initial amount obtained for processing. The anode sludge is easily separated from the electrolyte solution by filtration (unlike the anode sludge obtained by direct current in galvanostatic mode, other things being equal): 90% of anode sludge particles are between 146 to 510 µm in size. The resulting anode sludge can be processed using well-known methods [30], such as ammonia leaching with rhenium, tungsten, and molybdenum transfer into a solution, followed by the

Table 1. Influence of current program parameters on the anodic dissolution rate of GS32-VI alloy

Parameter	Value	Nickel concentration, C(Ni), g/dm^3	Dissolution rate of the alloy, g/h·cm^2	Current yield of nickel, η(Ni), %
Current pulse duration—500 ms, pause time between pulses—250 ms				
Amplitude of current pulse, A	0.5	9.75	0.027	60.5
	1.0	9.33	0.038	60.1
	1.5	10.20	0.042	62.5
	2.0	14.05	0.048	61.8
	2.5	15.12	0.054	57.2
Amplitude of current pulse—2.0 A, pause time between pulses—250 ms				
Current pulse duration, ms	200	9.90	0.020	61.,8
	500	10.44	0.038	63.8
	700	11.03	0.038	59.4
	900	12.34	0.044	53.2
	1200	14.75	0.048	49.9
Current pulse amplitude—2.0 A, current pulse duration—500 ms				
Pause time between pulses, ms	150	9.75	0.028	59.5
	250	9.33	0.036	61,1
	350	10.20	0.026	60.5
	450	14.05	0.020	56.8
	500	15.12	0.015	52.2

Table 2. Chemical composition of cathode deposit and anode sludge obtained during the anodic dissolution of GS32-VI alloy

Elements	Cathode deposit		Anode sludge	
	g	%	g	%
Ni	46.58	84.57	6.96	25.49
Co	7.20	13.07	0.54	1.98
Cr	1.03	1.87	1.43	5.23
Re	0.22	0.39	3.01	11.02
Al	0.05	0.10	0.49	1.79
Nb	–	–	1.59	5.82
Mo	–	–	1.05	3.84
Ta	–	–	3.88	14,20
W	–	–	8.37	30.63

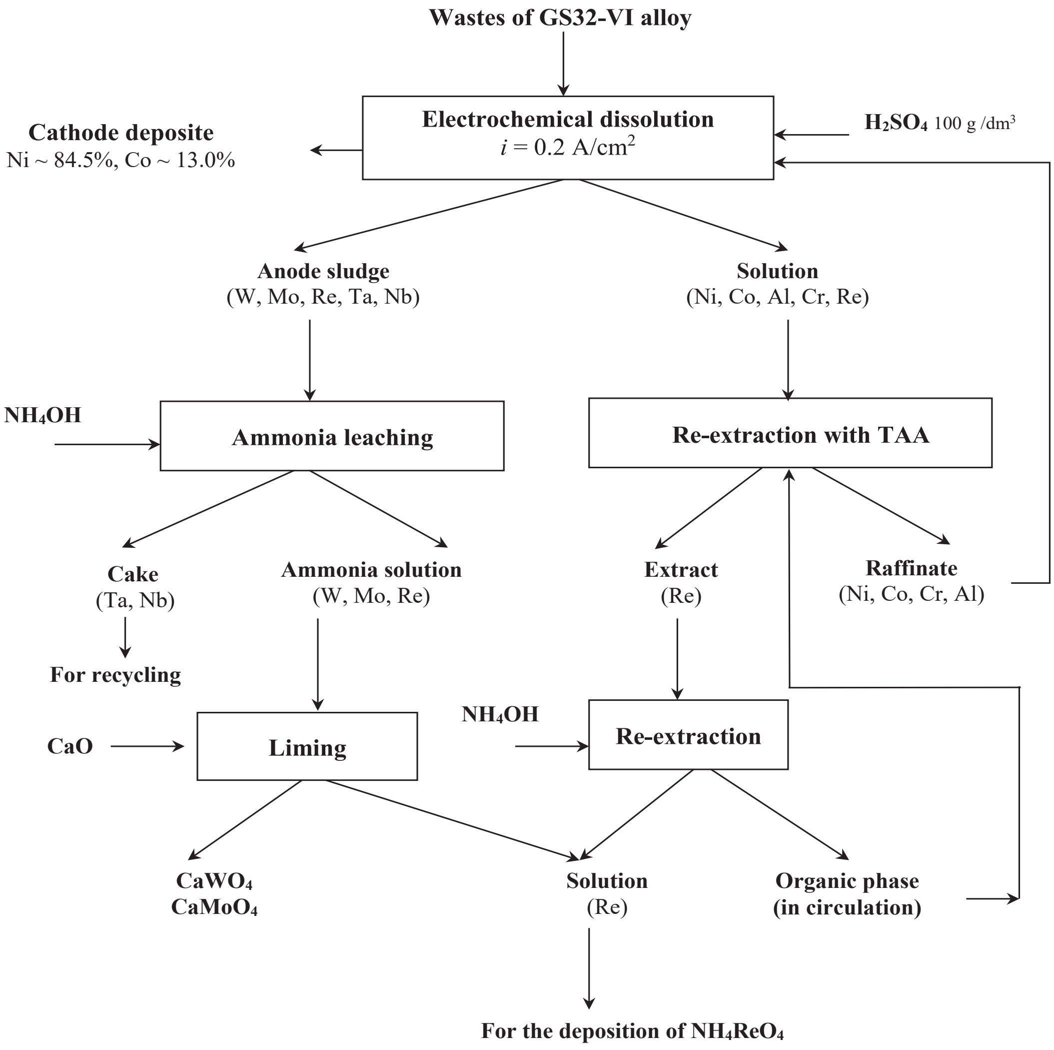

Fig. 7. The basic technological processing scheme for the heat-resistant GS32-VI alloy.

Figure 7. Schematic technological scheme of processing of heat-resistant alloy *HR32-VI*

deposition of artificial scheelite and the formation of a rhenium-containing solution, from which another commercial product is obtained—ammonium perrhenate (or potassium).

The total content of nickel and cobalt in the cathode deposit is 97.5%.

The basic technological scheme for the complex processing of the heat-resistant GS32-VI alloy is proposed based on the research (Fig. 7).

CONCLUSIONS

1. The effect of pulse current parameters (amplitude, pulse duration, and pause between pulses) on the electrochemical dissolution rate of GC32-VI alloy was shown. It was found that the current pulse amplitude was 2 A (with an anode area of $S = 10$ cm^2), the current pulse duration was 500 ms, the pause between pulses was 250 ms, the maximum dissolution rate of the alloy was 0.048 g/h·cm^2; the current output for nickel was 61.6% with the current program.

2. During the electrochemical dissolution of GC32-VI alloy by pulsed current, the optimal dissolution rate ratio of the alloy components was achieved, ensuring the production of a cathode precipitate with a total nickel and cobalt content of 97.5%.

3. The basic technological scheme for processing GS32-VI alloy was proposed.

Authors' contribution

All authors equally contributed to the research work.

The authors declare no conflicts of interest.

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