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Research article
Научная статья

Oxidative leaching of rhenium from grinding waste of rhenium-containing superalloys

I.E. Targanov, M.A. Solodovnikov, I.D. Troshkina

Mendeleev University of Chemical Technology of Russia
9 Miusskaya Sq., Moscow, 125047, Russia

✉ Irina D. Troshkina (troshkina.i.d@muctr.ru)

Abstract: The study investigated the feasibility of oxidative leaching rhenium in the presence of hydrochloric acid from machining waste (grinding waste) derived from products made of ZhS-32VI, a nickel-based heat-resistant alloy containing rhenium. This was achieved through agitation leaching process. The grinding waste fraction size of –0.071 mm, which accounted for the highest yield (49.2 wt.%), was utilized in the experiments. The rhenium leaching process was conducted in two variations: in the first option, grinding waste was mixed with a hydrochloric acid solution at ~100 °C, followed by the addition of hydrogen peroxide to the leaching solution after it had cooled; in the second option, leaching was performed using a hydrochloric acid solution with the gradual addition of hydrogen peroxide solution. The highest degree of rhenium leaching (91.0 %) was achieved in the first option. In this case, the initial concentration of hydrochloric acid was 8M, and the molar ratio of the added reagents was $\nu(HCl)$: $\nu(H_2O_2) = 2.7 : 1.0$. The kinetics of nickel leaching using a 6M hydrochloric acid solution at 70 °C, with a solid-to-liquid phase ratio of 1 g : 50 mL, was also examined. The analysis of the kinetic data, processed using the "contracting sphere," Ginstling–Brounshtein, and Kazeev–Erofeev models, indicates that the nickel leaching process occurs within the kinetic region. Additionally, the kinetics of rhenium leaching from the solid residue obtained after the hydrochloric acid leaching of nickel from grinding waste was investigated. Employing the same kinetic models to analyze the data, it was determined that the limiting stage of this process involves the diffusion of hydrogen peroxide within the rhenium-containing solid residue.

Keywords: rhenium, nickel, superalloys, grinding waste, leaching, hydrochloric acid, oxidizing agent, kinetics.

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Окислительное выщелачивание рения из шлифотходов ренийсодержащих суперсплавов

И.Е. Тарганов, М.А. Солодовников, И.Д. Трошкина

Российский химико-технологический университет им. Д.И. Менделеева
125047, Россия, г. Москва, Миусская пл., 9

✉ Ирина Дмитриевна Трошкина (troshkina.i.d@muctr.ru)

Аннотация: В агитационном режиме исследована возможность окислительного выщелачивания рения в присутствии соляной кислоты из отходов механической обработки (шлифотходов) изделий из Re-содержащего жаропрочного сплава ЖС-32ВИ на основе никеля. Использовали фракцию шлифотходов –0,071 мм с наибольшим выходом (49,2 мас.%). Процесс извлечения рения осуществляли в двух вариантах: в первом – шлифотходы контактировали с раствором соляной кислоты при температуре ~100 °С, после охлаждения раствора выщелачивания в него добавляли раствор пероксида водорода; во втором – выщелачивание проводили с применением раствора соляной кислоты с порционным добавлением раствора пероксида водорода. Наибольшее значение степени извлечения рения (91,0 %) наблюдалось при выщелачивании в первом варианте, начальная концентрация соляной кислоты составила 8М, мольное соотношение добавляемых реагентов – $\nu(HCl) : \nu(H_2O_2) = 2,7 : 1,0$. Была изучена кинетика выщелачивания никеля раствором соляной кислоты (6М) при температуре 70 °С и соотношении фаз шлифотход : раствор, равном 1 г : 50 мл. Анализ обработки кинетических данных с использованием моделей «сжимающейся сферы», Гинстлинга–Броунштейна и Казеева–Ерофеева позволяет утверждать, что процесс выщелачивания никеля протекает в кинетической области. Исследована кинетика выщелачивания рения из твердого остатка солянокислого выщелачивания никеля из шлифотходов. Применение для обработки данных тех же кинетических моделей позволяет выделить диффузию пероксида водорода в ренийсодержащем твердом остатке как лимитирующую стадию.

Ключевые слова: рений, никель, суперсплавы, шлифотходы, выщелачивание, соляная кислота, окислитель, кинетика.

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Introduction

The annually increasing global demand for high-tech materials, including rare elements, underscores the importance of recycling secondary raw materials. One such rare element is rhenium, which is extracted from primary raw materials solely as a by-product [1; 2]. Rhenium's main natural sources encompass sulfide molybdenum and copper ores, polymetallic uranium ores, and the fumarole gases of the Kudryavy Volcano on Iturup Island [3]. Depletion projections for rhenium reserves are on par with those for other non-ferrous and rare metals, whose resources are anticipated to be depleted within the next 100—110 years [2]. Consequently, it is imperative to develop and enhance recycling technologies for rhenium.

According to forecasts [4], the demand for rhenium in Russia is expected to reach 20 tons/year by 2032 and 35 tons/year by 2064.

The primary utilization of rhenium (78 %) occurs in the production of superalloys used for aircraft jet engines and turbines to generate electricity, where rhenium plays a vital role as an alloying element [5; 6]. Nickel-based superalloys exhibit exceptional oxidation resistance and mechanical strength. Substantial enhancements in their mechanical properties at high temperatures have been achieved through the careful control of material structure, incorporating alloying elements like rhenium, ruthenium, and hafnium [7; 8]. The addition of Re to these superalloys creates solid solution strengthening of the matrix, significantly improving heat resistance. However, it's worth noting that the cost of adding rhenium, for example, at 3 wt.% in the CMSX-4 alloy, accounts for approximately 60 % of the alloy's total cost [9; 10].

Monocrystalline rhenium-containing superalloys based on nickel are predominantly employed [6; 10—12].

The total waste generated from rhenium-containing heat-resistant nickel alloys, primarily consisting of discarded parts from gas turbine engines, was estimated to be 25—35 tons/year in the Russian Federation in 2012 [13]. Assuming a rhenium content of 3 %, this amounts to 0.75—1.0 tons/year of recoverable rhenium.

Existing technologies for processing waste form nickel-based heat-resistant alloys can be categorized into two groups: pyrometallurgical and hydrometallurgical [6; 14; 15]. Pyrometallurgical methods encompass direct waste melting and oxidative thermal methods [16], while hydrometallurgical approaches are based on electrochemical processes (anodic dissolution) [12; 17] and methods involving the decomposition of waste in acid solutions. The choice of processing technology depends on the type of raw material (solid parts or waste from mechanical processing, such as grinding waste) and the availability of specialized equipment like vacuum furnaces and electrolysis cells, among others.

Methods based on waste decomposition using acid solutions can involve both the removal of the alloy base and direct oxidative leaching to extract rhenium into solution. Leaching agents like mineral acids and their mixtures, in the presence of an oxidizing agent, can be employed for efficient rhenium leaching. Nitrate processing of multi-component alloys has proven effective [14], although the presence of nitrate ions in solutions can complicate subsequent sorption of rhenium. In a separate study [18], rhenium was leached using "aqua regia". When sulfuric acid is used, it becomes possible to leach the alloy base, nickel [19], followed by the addition of an oxidizing agent to isolate rhenium, or by using a mixture of sulfuric acid with an oxidizing agent like hydrogen peroxide for direct rhenium extraction into solution [14].

The high content of nickel and cobalt in grinding waste, both valuable non-ferrous metals, justifies the use of hydrochloric acid leaching during their comprehensive processing. In chloride environments, it's possible to separate these elements due to their similar chemical properties, requiring fewer steps in the preparation of the leaching solution [20].

The objective of this study is to establish the patterns of oxidative leaching of rhenium using hydrochloric acid solutions from grinding waste derived from a rhenium-containing nickel-based superalloy.

Research methodology

The focus of this study was the grinding waste derived from the ZhS32-VI superalloy, which exhibits the following elemental composition, wt.%: 1.5 Re; 9.0 Co; 8.5 W; 5.9 Al; 4.9 Cr; 4.0 Ta; 1.6 Nb; 1.0 Mo; 0.15 C; 0.02 V; 0.025 Ce, 60.0 Ni. The particle size distribution of this waste material is summarized in Table 1.

The grinding waste with the highest weight fraction (49.2 wt.%), specifically with a particle size of –0.071 mm (as shown in Table 1), was selected for use in the study.

The choice of this finer particle size is based on its larger specific surface area and higher mass yield, making it a preferred option for selecting a hydrometallurgical processing method. This method includes the decomposition of waste using hydrochloric acid solutions, in the presence of an oxidizing agent, specifically a 30 % H_2O_2 solution.

In order to ensure the purity of the grinding waste and eliminate contaminants such as glycol, mineral oils, lubricating additives, surfactants, corrosion inhibitors, and rags, which may be present due to the use of various lubricants and coolants during machining workpieces, the grinding waste from rhenium-containing superalloys underwent a cleaning process. This involved washing the waste with hot water (~90 °C) followed by drying prior to conducting the experiments.

A sample of the grinding waste was brought into contact with a hydrochloric acid solution at various phase ratios of solid to liquid, ranging from 1 : 50 to 1 : 100 (g : mL), under agitation. A mixing speed of 200 min^{-1} was selected, considering that the grinding waste particles were suspended and thoroughly mixed while ensuring a continuous flow of the solution around them. Depending on the specific experiment, the reaction mixture was heated and leached at a temperature of 100 °C for 60 minutes. After cooling the reaction mixture, hydrogen peroxide solution (*I*) was added to it batchwise. The experimental leaching setup consisted of a flask heater containing a three-neck flask equipped with a reflux condenser, and mixing was facilitated using an overhead stirrer.

Table 1. **Particle size distribution of grinding waste of ZhS32-VI superalloy**

Таблица 1. Фракционный состав шлифотходов суперсплава ЖС32-ВИ

Particle size, mm	Yield, wt.%
+2.0	3.1
–2.0+1.2	0.7
–1.2+1.0	2.0
–1+0.5	1.1
–0.5+0.1	24.7
–0.1+0.071	19.2
–0.071	49.2

When conducting experiments without heating to prevent spontaneous heating and potential hydrogen peroxide decomposition, the process took place within a thermostatically controlled jacketed cell that was actively cooled using running water. The oxidizing agent was added to the cooled reaction mixture batchwise, following each hour of the experiment (*II*). Stirring was maintained at a speed of 200 min^{-1}. Periodically, throughout the experiment, samples of the solution were withdrawn and subjected to analysis for rhenium content using the photometric method with ammonium thiocyanate. In order to ensure an adequate concentration of hydrochloric acid (with at least a 30 % excess), the selection was made considering theoretical calculations based on the chemical reactions of the acid with the constituent metals of the alloy and those components of the alloy that, as indicated in published data, are reactive with it.

The kinetics of nickel leaching from grinding waste using a hydrochloric acid solution was investigated under agitation in a thermostated reactor for a duration of 15 h at 70 °C. A sample of grinding waste was brought into contact with a 6M hydrochloric acid solution at a solid to liquid phase ratio of 1 : 50 (g : mL). At specific time intervals, samples of the solution were obtained and analyzed for nickel content using the titrimetric method with EDTA-Na_2.

The kinetics of rhenium leaching from the concentrate formed after the removal of nickel from waste using a solution of hydrochloric acid and hydrogen peroxide was investigated under agitation mode. The experiments were conducted in a thermostatically controlled cell that was actively cooled by running water. A sample of rhenium concentrate was brought into contact with a hydrochloric acid solution initially at a concentration of 2M, with a solid-to-liquid phase ratio of 1 : 250 (g : mL). The oxidizing agent was added in 1 ml increments every 30 min. At specific time intervals, samples of the solution were extracted and subjected to analyzed for Re content.

The degree of saturation of the solution with rhenium (or nickel), $\alpha_{(Me)}$, was calculated using the following equation:

$$\alpha_{(Me)} = C_{(Me)}V_{solution} / (\omega_{(Me)}m_{sample}),$$

where $C_{(Me)}$ represents the current concentration of the metal (rhenium or nickel), g/L; $V_{solution}$ stands for the solution volume, L; $\omega_{(Me)}$ denotes the weight fraction of the metal (rhenium or nickel) in grinding waste; m_{sample} is the weight of the sample, g.

Results and discussion

Rhenium metal undergoes a reaction with hydrogen peroxide to produce rhenium acid as described by the following equation:

$$2Re + 7H_2O_2 \rightarrow 2HReO_4 + 6H_2O.$$

In preliminary experiments, it was determined that quantitatively extracting rhenium into solution is not achievable solely by using a solution of hydrogen peroxide, without first removing the nickel component, which is a fundamental constituent of the superalloy.

Table 2 provides a summary of the data pertaining to the oxidative leaching of rhenium from pre-washed grinding waste originating from a rhenium-containing superalloy. These leaching experiments were conducted in hydrochloric acid solutions with the aid of hydrogen peroxide as an oxidizing agent, under various leaching methods and conditions.

The most effective extraction of rhenium into solution occurs when the waste is heated in an acidic solution with the addition of an oxidizing agent after the mixture has been cooled to room temperature. It appears that the initial removal of the majority of the alloy by preheating it in hydrochloric acid solutions a temperature of 100 °C for 1 hour facilitates better access of the oxidizing agent to the rhenium within the alloy.

Furthermore, elevating the concentration of the hydrochloric acid solution from 6M to 8M during the preheating step and maintaining it during the leaching process results in a 7.5 % increase in the degree of rhenium extraction into solution, followed by the subsequent addition of an oxidizing agent.

The extent of nickel leaching, which constitutes the base of the superalloy, when treated with a 6M hydrochloric acid solution at a temperature of 70 °C over time, is graphically depicted in Fig. 1.

As illustrated in in Fig. 1, it’s evident that equilibrium is achieved after ~600 min.

In order to pinpoint the limiting stage of the nickel leaching process with the hydrochloric acid solution, the kinetic data were subjected to analysis using both kinetic and diffusion mathematical models (Fig. 2, Table 3).

The “contracting sphere” model, also known as the Gray—Weddington equation [22], is used to describe processes where a reaction takes place on the surface of a spherical solid that loses mass and diminishes in size

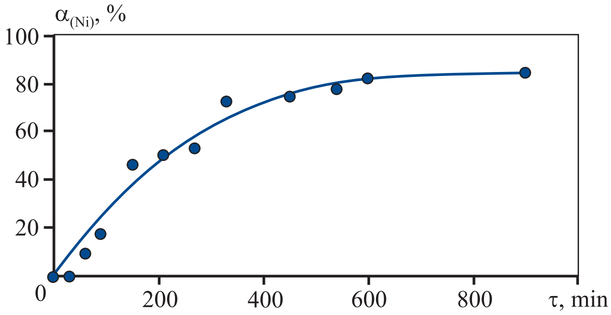

Fig. 1. Temporal variation in nickel leaching using a 6M hydrochloric acid solution at 70 °C

Рис. 1. Зависимость степени выщелачивания никеля солянокислым раствором (6M) при температуре 70 °C от времени

Table 2. **Hydrochloric acid oxidative leaching of rhenium**

Таблица 2. Солянокислое окислительное выщелачивание рения

Initial concentration of HCl	Procedure conditions	Mole ratio $\nu(HCl) : \nu(H_2O_2)$	Phase ration, g : mL	$\alpha_{(Re)}$, %
8M	*I*	2.7 : 1.0	1 : 130	91.0
6M	*I*	2.0 : 1.0	1 : 130	83.5
6M	*I*	1.4 : 1.0	1 : 50	71.4
6M	*II*	2.4 : 1.0	1 : 100	68.5
8M	*II*	6.6 : 1.0	1 : 50	47.0

Table 3. **Kinetic characteristics of hydrochloric acid leaching of nickel from grinding waste obtained using various models**

Таблица 3. Кинетические характеристики солянокислого выщелачивания никеля из шлифотходов, полученные при использовании различных моделей

Equation of “contracting sphere”		Ginstling–Brounshtein equation		Kazeev–Erofeev equation	
$1 - (1 - \alpha)^{1/3} = k\tau$		$1 - (2/3)\alpha - (1 - \alpha)^{2/3} = k\tau$		$\ln[-\ln(1 - \alpha)] = n\ln\tau + \ln k_\tau$	
$k \cdot 10^4$, min^{-1}	R^2	$k \cdot 10^4$, min^{-1}	R^2	n	R^2
8	0.933	3	0.883	1.56	0.849

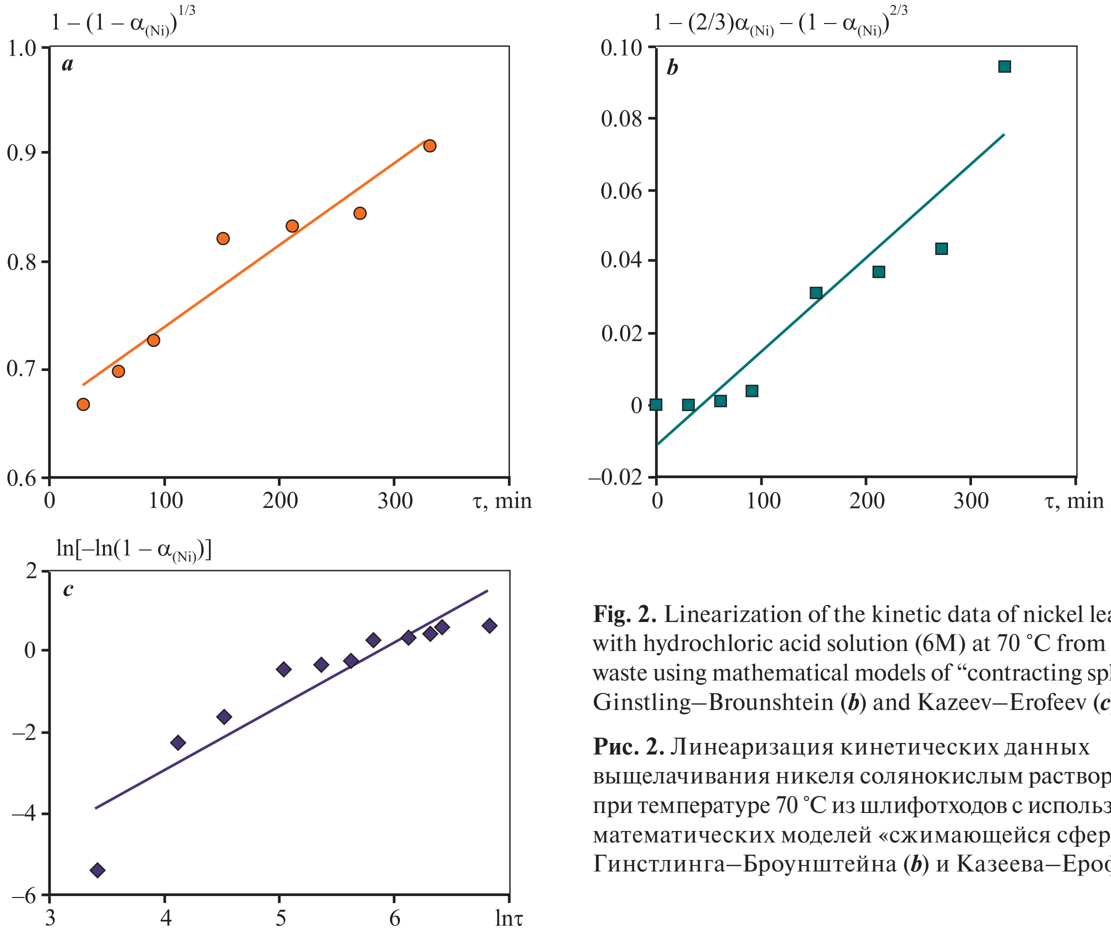

Fig. 2. Linearization of the kinetic data of nickel leaching with hydrochloric acid solution (6M) at 70 °C from grinding waste using mathematical models of “contracting sphere” (***a***), Ginstling–Brounshtein (***b***) and Kazeev–Erofeev (***c***)

Рис. 2. Линеаризация кинетических данных выщелачивания никеля солянокислым раствором (6M) при температуре 70 °C из шлифотходов с использованием математических моделей «сжимающейся сферы» (***a***), Гинстлинга–Броунштейна (***b***) и Казеева–Ерофеева (***c***)

during the reaction, leading to the formation of an undissolved porous layer of product. This equation is particularly suitable for processes occurring within the kinetic range of reactions and is expressed as follows:

$$1 - (1 - \alpha)^{1/3} = k\tau, \qquad (1)$$

where α represents the leaching rate (as a fraction), k is the rate constant of the reaction, min^{-1}, and τ is the time, min.

In cases where a dense, non-porous product layer is formed, the Ginstling—Brounshtein equation is employed [20, 22]:

$$1 - (2/3)\alpha - (1 - \alpha)^{2/3} = k\tau. \qquad (2)$$

The use of the generalized kinetic Kazeev—Erofeev equation helps determine the limiting stage of the process and estimate the values of the n index in the equation:

$$\alpha = 1 - \exp(-K\tau^n), \qquad (3)$$

where K and n are the kinetic parameters. This equation serves as a function of the likelihood of the degree of transformation, applicable to both topochemical reactions and reactions of different types.

In its doubly logarithmic form, Eq. (3) is linearized as follows:

$$\ln[-\ln(1-\alpha)] = n\ln\tau + \ln k_\tau. \quad (4)$$

Based on the results of the mathematical analysis of kinetic data for the leaching of nickel from the grinding waste of rhenium-containing superalloys, it is evident that the data are more suitably linearized by employing the “contracting sphere” equation. Furthermore, the examination of mathematical processing using the Kazeev—Erofeev equation has allowed us to ascertain that the value of the $n = 1.56$. This value indicates that the leaching of nickel follows a kinetic reaction in the process.

Regarding the leaching of rhenium from the concentrate formed after the removal of nickel, which is the primary component of the superalloy, this process was conducted using a solution of hydrogen peroxide in the presence of hydrochloric acid. The degree of rhenium leaching from the concentrate, with the addition of a 2M hydrochloric acid solution and periodic introduction of hydrogen peroxide (Fig. 3), exhibits a characteristic convex shape over time and reaches a plateau within ~1 h.

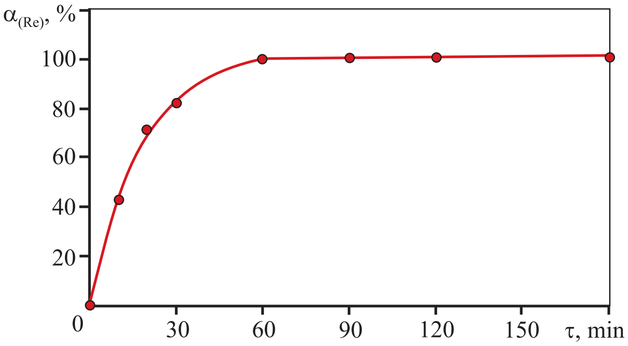

Fig. 3. Temporal variation of rhenium leaching using hydrogen peroxide (30 %) in the presence of hydrochloric acid (2M)

Рис. 3. Зависимость степени выщелачивания рения пероксидом водорода (30 %) в присутствии соляной кислоты (2M) от времени

The degree of rhenium leaching rate into the solution achieved a level of ≥99 %.

In order to identify the rate-limiting stage of the process, the kinetic data were subjected to analysis using the previously described kinetic and diffusion mathematical models (Fig. 4, Table 4).

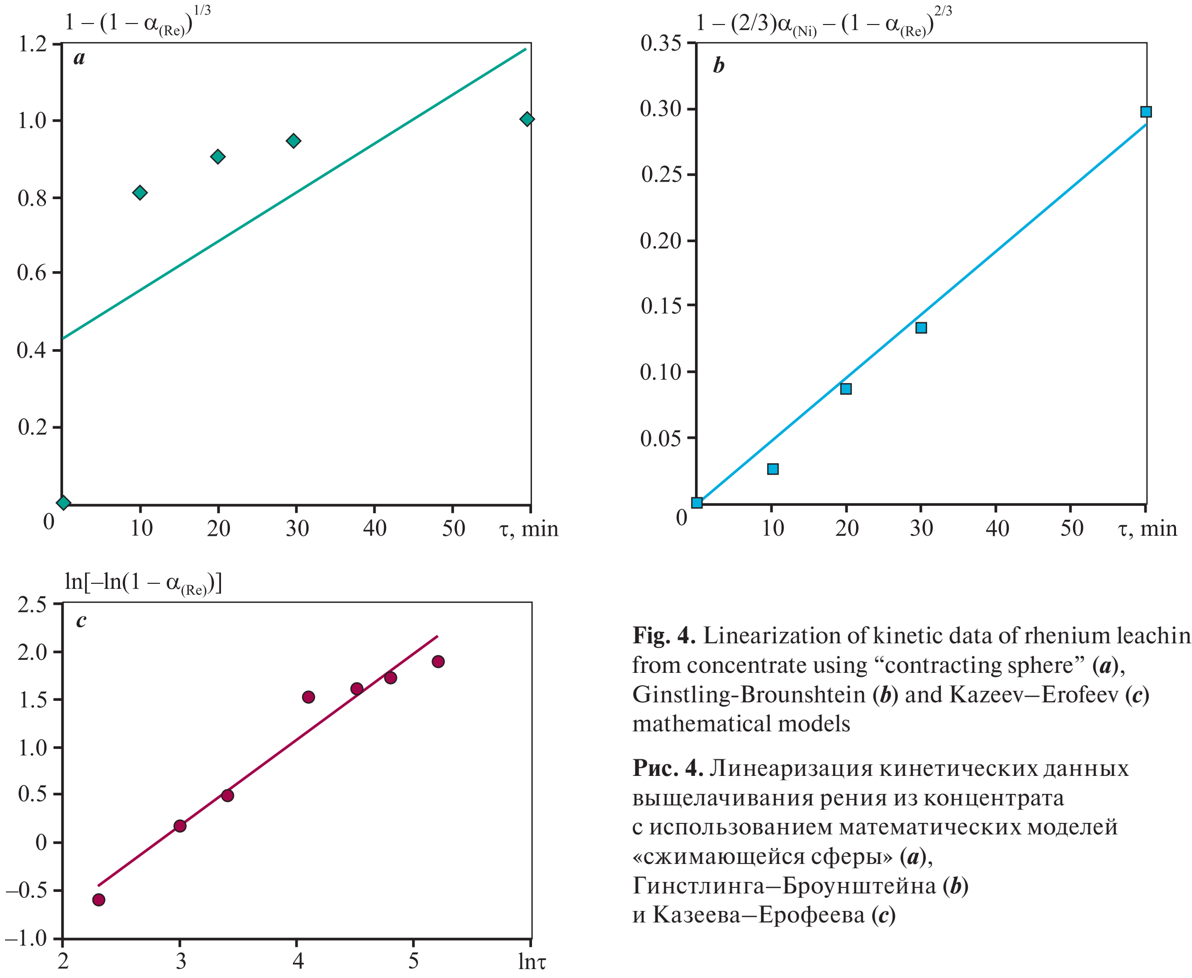

Fig. 4. Linearization of kinetic data of rhenium leaching from concentrate using “contracting sphere” (***a***), Ginstling-Brounshtein (***b***) and Kazeev—Erofeev (***c***) mathematical models

Рис. 4. Линеаризация кинетических данных выщелачивания рения из концентрата с использованием математических моделей «сжимающейся сферы» (***a***), Гинстлинга—Броунштейна (***b***) и Казеева—Ерофеева (***c***)

Table 4. **Kinetic characteristics of rhenium leaching from concentrates obtained using different models**

Таблица 4. Кинетические характеристики выщелачивания рения из концентрата,
полученные при использовании различных моделей

Equation of “contracting sphere”		Ginstling–Brounshtein equation		Kazeev–Erofeev equation	
$1-(1-\alpha)^{1/3}=k\tau$		$1-(2/3)\alpha-(1-\alpha)^{2/3}=k\tau$		$\ln[-\ln(1-\alpha)]=n\ln\tau+\ln k_\tau$	
$k\cdot 10^2$, min^{-1}	R^2	$k\cdot 10^3$, min^{-1}	R^2	n	R^2
1.2	0.488	4.7	0.986	0.9	0.958

The kinetic data concerning the leaching of rhenium from the concentrate formed after the removal of nickel, with a substantial coefficient of determination, have been successfully linearized using the Ginstling—Brounshtein equation. This equation is particularly relevant for describing processes that occur in the diffusion response region. The value of the index n, which was obtained during the processing of kinetic data using the Kazeev—Erofeev equation, was found to be less than 1. This observation corroborates the results of the analysis conducted using the Ginstling—Brounshtein equation, affirming that the limiting stage of the process is indeed diffusion-driven.

Conclusion

The experimental study of the oxidative hydrochloric acid leaching of rhenium from superalloy grinding waste (particle size fraction –0.071 mm) has yielded significant findings. The highest degree of rhenium leaching (α = 91.0 %) was achieved when the process involved preliminary mixing of grinding waste in an 8M HCl solution for 1 h at approximately 100 °C. Subsequently, the reaction mixture was cooled, and hydrogen peroxide was added batchwise. This study demonstrated that increasing the concentration of hydrochloric acid from 6M to 8M, under the selected conditions, resulted in a 7.5 % increase in the degree of rhenium extraction into solution.

The analysis of kinetic data regarding the leaching of nickel, the primary component of the superalloy, from grinding waste with a 6M hydrochloric acid solution at 70 °C, using the “contracting sphere”, Ginstling—Brounshtein, and Kazeev—Erofeev mathematical equations, reveals that the process operates within the kinetic reaction range.

Furthermore, the analysis of the mathematical processing of kinetic data for rhenium leaching using a solution of hydrogen peroxide in hydrochloric acid (2M) from a concentrate, which is the solid residue obtained after the leaching of nickel from grinding waste with hydrochloric acid, employing diffusion and kinetic models, indicates that the leaching rate is predominantly limited by the diffusion of hydrogen peroxide within the solid concentrate.

Subsequent processing of the leaching solutions through solvent extraction allows for the isolation of ammonium perrhenate. Rhenium metal powder, obtained from this compound through hydrogen reduction, can be employed in the production of various alloys.

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Information about the authors

Igor E. Targanov – Postgraduate Student of the Department of Technology of Rare Elements and Nanomaterials (TREN), Mendeleev University of Chemical Technology of Russia (MUCTR).
https://orcid.org/0000-0001-8354-0018
E-mail: targanov.igor@yandex.ru

Maksim A. Solodovnikov – Student of the Department of TREN, MUCTR.
https://orcid.org/0009-0009-3497-160X
E-mail: solodovnikovmaksim1@gmail.ru

Irina D. Troshkina – Dr. Sci. (Eng.), Professor of the Department of TREN, MUCTR.
https://orcid.org/0000-0002-5523-0247
E-mail: troshkina.i.d@muctr.ru

Информация об авторах

Игорь Евгеньевич Тарганов – аспирант кафедры технологии редких элементов и наноматериалов (ТРЭН) Российского химико-технологического университета (РХТУ) им. Д.И. Менделеева.
https://orcid.org/0000-0001-8354-0018
E-mail: targanov.igor@yandex.ru

Максим Александрович Солодовников – студент кафедры ТРЭН РХТУ им. Д.И. Менделеева.
https://orcid.org/0009-0009-3497-160X
E-mail: solodovnikovmaksim1@gmail.ru

Ирина Дмитриевна Трошкина – д.т.н., профессор кафедры ТРЭН РХТУ им. Д.И. Менделеева.
https://orcid.org/0000-0002-5523-0247
E-mail: troshkina.i.d@muctr.ru

Contribution of the authors

I.E. Targanov – conducted experiments, contributed to the discussion of the results, authored the manuscript.

M.A. Solodovnikov – analyzed samples through photometric and titrimetric methods, contributed to the discussion of the results.

I.D. Troshkina – formulated the research objectives, contributed to the discussion of the results, revised the manuscript.

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