A heat-resistant superalloy from destroyed special equipment was used for further processing to extract valuable metals such as rhenium, nickel, cobalt, tungsten, molybdenum, niobium, tantalum, and others. The need to develop an effective method for the electrochemical dissolution of this superalloy is due to the shortage and high cost of the component metals, especially rhenium. The electrochemical dissolution method is effective for the rapid and complete dissolution of such hard alloys, optimizing the extraction process of valuable components.

In the course of the work, the composition of the unknown superalloy was determined, and the possible grade of the alloy was identified as JS32-VI. For the first time, a comparison of the anodic behavior of the heat-resistant superalloy containing rhenium in various electrolyte solutions, including methanesulfonic acid, was carried out. This comparison helped determine which electrolyte is best suited for dissolving the superalloy and extracting valuable metals from it.

The results showed that solutions containing chloride ions (NaCl and HCl) are the most effective for the electrochemical dissolution of the superalloy. These results are explained by the fact that chloride ions help remove the passive oxide film from the metal surface. Thus, chloride solutions provide more effective dissolution of the superalloy compared to methanesulfonic acid and sulfuric acid.

The key finding of the study is the identification of chloride solutions as the most effective for dissolving the superalloy, which optimizes the process of extracting valuable metals. The application of these methods will contribute to resource conservation and the reduction of production costs, which is important for industries using such materials

Keywords: turbine elements, electrochemical dissolution, methanesulfonic acid, hydrochloric acid, sodium chloride, sulfuric acid

DETERMINATION OF PROCESSING CONDITIONS FOR A HEAT-RESISTANT SUPERALLOY USED IN TURBINE ELEMENTS

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1. Introduction

Metals and their alloys have become an integral part of engineering constructions, devices, installations, and components, without which modern life is impossible. For example, modern special alloys on average contain up to five metals, with the most common components being Cu, Co, Ni, Ti, Ta, W, Cr, Sn, Mn, Al, rare earth metals [1], and in some cases, precious metals. However, many of the metals in multicomponent alloys are often rare, expensive, have limited reserves, or are not available as deposits in Ukraine [2].

One of the methods for efficient resource utilization is the recycling of alloy scrap to extract valuable components. Ideally, this process should be fast while minimizing energy consumption. Electrochemical dissolution could be a solution to these challenges. However, combined electrochemical and chemical alloy recycling offers greater flexibility in separating components. It is important to note that the extraction of metals at the end of the process can result in either metal compounds (mostly salts) or pure metals. When the goal is to obtain metals in their pure state, electrochemical methods can achieve high purity levels. High purity is crucial for the final physical and chemical properties of the metals. For example, copper with 99.9999 % purity has significantly higher electrical conductivity compared to copper with 99.9 % purity. The reason for this is impurities, which cause defects in the crystal lattice, leading to reduced conductivity [3].

It is also important to note that the form of electrochemically deposited alloy components affects the product's value. For example, fine powder fractions of metals can be tens or

even hundreds of times more expensive than the same metals in their regular form, due to their specialized applications [4, 5]. Electrochemical deposition methods can also meet this demand by synthesizing metals in powdered form [6, 7].

Thus, recycling alloys while determining optimal conditions for process acceleration is critically important for the country's economy.

2. Literature review and problem statement

Electrochemical methods allow for fast and efficient dissolution of scrap, metal refining, and obtaining various metal forms (powder or solid metal). After electrochemical dissolution as the first stage of processing, the components of the alloy can be effectively separated using combined methods. However, if the first stage of processing involves dissolving a superalloy in acids or using pyrometallurgical methods, the metal must be broken into small pieces. This presents certain challenges, as multicomponent superalloys used in turbine elements are extremely hard [8, 9]. Studies have shown that the problem of substituting rhenium with other elements to achieve similar creep resistance remains unresolved. On the other hand, pyrometallurgical methods require elevated temperatures, increasing the cost of the recycling process. Thus, a logical approach would be to use the following generalized scheme: first, electrochemical dissolution, followed by separation of elements through specific reactions, and finally, electrochemical deposition (electrochemical refining, electroextraction) or deposition in the form of insoluble substances through chemical or electrochemical methods.

However, issues related to the recycling of various superalloy grades remain unresolved [8, 9]. This is due to their differing compositions and, consequently, their different physical and chemical properties, meaning there are no universal conditions, modes, or solutions for their electrochemical dissolution.

Nevertheless, recycling is essential since heat-resistant superalloys contain valuable metals, including Fe, Ni, Co, Cr, W, Mo, Ta, Ti, Al, and Re [10]. This study emphasizes the need for further research to understand unresolved issues regarding the detailed correlation between alloy microstructure and mechanical properties, as well as processing based on these properties. These elements significantly improve the chemical and mechanical properties of materials, including hardness, creep resistance, and corrosion resistance at high temperatures. Special attention should be given to Re, a key element in these alloys. Rhenium is rare in the Earth's crust, with only a few known deposits worldwide, and its annual production does not exceed a few dozen tons [11], while its market price approaches 2500 USD/kg [12]. This highlights that recycling scrap from such alloys can not only yield valuable materials but is also a strategically important industry.

In [13], the authors processed spent lithium power sources and nickel-based superalloy powder, with $Mn(ReO_4)_2 \cdot 2H_2O$ as the final product. The alloy was dissolved in a mixture of acids with an added oxidizer. The research results demonstrate effective recycling, but the need to grind the scrap into powder increases the method's cost. However, the presented results apply to alloy powder and do not address the recycling of large-scale superalloy scrap.

In [14], the recycling of the CMSX-4 superalloy containing nickel and rhenium was also demonstrated. Rhenium was extracted using a two-stage leaching process with organic reagents. Similar to the previous study, the research results do not resolve the issue of processing materials beyond those

with high specific surface areas, and the need for heating further increases recycling costs.

The study [15] shows a successful example of selective electrochemical recycling of the superalloy VNZh90, containing nickel, iron, and tungsten. The process took place at room temperature without the need to grind the alloy, confirming the feasibility of using electrochemical methods for large alloy fragments. However, the alloy used in the study differs significantly in composition from heat-resistant rhenium-containing alloys, so the conditions and chosen electrolyte do not resolve the issue of their electrochemical dissolution.

The study [16] focuses on the recycling of superalloys containing only W and Re. Rhenium was extracted using a pyrometallurgical method, first oxidizing with air at 675 °C to form volatile Re_2O_7, which was then absorbed by an NH_4OH solution to form NH_4ReO_4. However, this method is limited to two-component alloys, making it less versatile.

In [17], the extraction of rhenium was proposed using a molten mixture of 70-80 % NaOH and 20-30 % Na_2SO_4 with $NaNO_3$, $K_2S_2O_8$, and Na_2O at temperatures of 800–1200 °C. Hydrometallurgical methods were then used to recover individual metals. Despite the potential for recovering individual metals, the high temperatures required significantly increase energy consumption, limiting the method's application.

One possible approach for full recycling of a multicomponent nickel-based alloy containing rhenium is the scheme proposed in [9]. According to this scheme, the alloy is first electrochemically dissolved in H_2SO_4, HCl, or HNO_3. Then, a combination of chemical and electrochemical methods is used to separate and recover the metals in the form of solutions, solid substances, or reduced metals. However, the authors do not propose using NaCl during electrochemical dissolution, which would be much cheaper than HCl.

The study [18] is dedicated to rhenium extraction through electrochemical dissolution in a mixture of HNO_3 and HCl acids from secondary materials, including superalloys, metallurgical residues, ores, and/or mixtures of secondary solid materials. The dissolution was carried out at potentials >500 mV (Ag/AgCl), resulting in a mixture of rhenium and other metal salts such as nickel and cobalt. Other metals remained in the solid leaching residue. Rhenium was separated from the leach solution by sulfide precipitation as Re_2S_7, absorption on activated carbon, absorption resin, or solvent extraction. The final product was expected to be NH_4ReO_4. This approach corresponds to the scheme proposed at the beginning of the section, being the least energy-intensive and fastest option.

A comparison of approaches to superalloy recycling [8–18] shows that the primary processing (transforming alloy elements into a soluble form) depends on the specific composition, characteristics, and size of the scrap. Electrochemical dissolution is more suitable for processing relatively large alloy fragments since it does not require high temperatures or grinding and, in some cases, allows for selective recycling. If the alloy's composition is unknown, the composition and feasibility of electrochemical recycling must first be determined. It is also essential to identify the most effective electrolytes, with selected solutions of the following substances: NaCl, HCl, H_2SO_4, and CH_3SO_3H. This selection is based solely on a rational approach. For example, chlorides, in the form of salts or acids, are used for effective dissolution in refining and electroplating processes during anode dissolution [19], as the Cl⁻ ion is a powerful depassivator 20]. Studies highlight that the mechanisms of protective film breakdown under high-temperature and chloride ion concentration conditions remain unresolved, which may influence the choice

of conditions during electrochemical dissolution. Meanwhile, NaCl, containing the Cl⁻ anion, is a relatively inexpensive reagent. Sulfuric acid is also widely used in metal dissolution and refining processes [21]. As an alternative, methanesulfonic acid can be used as an electrolyte, as it is highly soluble in water, has high electrical conductivity, and is used as an electrolyte for some electroplating processes [22, 23]. However, the possibility of using methanesulfonic acid as an electrolyte for the electrochemical dissolution of superalloys remains unresolved.

The analysis of the provided information suggests that it is reasonable to conduct research dedicated specifically to the electrochemical dissolution of nickel-based superalloys with rhenium as the first stage of recycling.

3. The aim and objectives of research

The research aims to determine the conditions for the initial stage of recycling – the electrochemical dissolution stage for a heat-resistant superalloy containing rhenium. This work will enable the identification of starting conditions for electrochemical dissolution as the first stage of complete recycling for a superalloy of unknown composition, to isolate individual components as pure metals or their compounds.

To achieve this aim, the following objectives were addressed:
– determine the composition of the studied alloy;
– establish the reactivity of the alloy during anodic dissolution in the selected electrolyte solutions.

4. Materials and methods

4. 1. The object and hypothesis of research

The object of the research is the kinetics of the processes occurring during the electrochemical dissolution of the selected superalloy.

The research hypothesis is the actual possibility of accelerated dissolution of the chosen superalloy. It is assumed that anodic dissolution in different electrolytes should exhibit significant differences in speed, as the presence of various ions affects the surface state of the dissolving metal in different ways.

As a simplification, it was assumed that during the relatively short time required to obtain cyclic amperometry curves, the morphology of the superalloy changes insignificantly. Therefore, it is considered that the calculated current density for a given current remains constant.

4. 2. Materials and reagents

Distilled water was used to prepare the solutions. NaCl, HCl, H_2SO_4, and CH_3SO_3H were used with analytical reagent grade purity.

Turbine blade fasteners made from an unknown-grade superalloy, sourced from destroyed military equipment during the battles in Ukraine, were used in the study (Fig. 1).

The surface of the turbine elements appears to be covered with corrosion marks, which may indicate that the metal was exposed to an oxidative environment at high temperatures.

a   *b*

Fig. 1. Turbine elements: *a* — U-shaped fastener; *b* — part of the turbine blade

4. 3. Methods for studying the superalloy characteristics

X-ray fluorescence analysis.

The composition of the superalloy was determined using the "SPRUT" spectrometer (Ukraine) [24].

Electrochemical experiments.

To determine the electrochemical properties, a potentiostat MTech SPG-500fast (Ukraine) was used. Graphite was employed as the counter electrode, and an Ag|AgCl electrode in 3.5 M KCl was used as the reference electrode. Cyclic voltammetry (CV) curves were recorded at a scan rate of 50 mV·s⁻¹ over a wide potential window to evaluate the electrochemical activity of the alloy. Before measuring the electrochemical characteristics, the electrode was polished with sandpaper (80 μm grit) and degreased. To record data with a known current density, part of the metal was exposed while the rest was insulated with PTFE tape.

Calculation of results.

To determine the total specific charge, Q, in mA·s·cm⁻² for anodic (metal or alloy dissolution) and cathodic processes, the obtained cyclic voltammetry curves (CVs) were integrated using a program written in Python (Python Software Foundation (PSF)). The program also utilized the open-source libraries Pandas (Python data analysis library) and Matplotlib (Python plotting library). The integration was performed separately for anodic and cathodic current densities using the trapezoidal rule. Two separate capacities were calculated for the anodic and cathodic charges. The conditions for separating the two charges were: 1 – a change in the polarity of the current density; 2 – a change in the direction of the potential sweep.

When either condition was met, the charge was reset, and the integration restarted. For example, in the CV (Fig. 2), four distinct shaded zones represent areas for which the charge would be calculated based on the specified conditions.

Thus, for this CV, four charges would be calculated: two for the anodic and two for the cathodic processes.
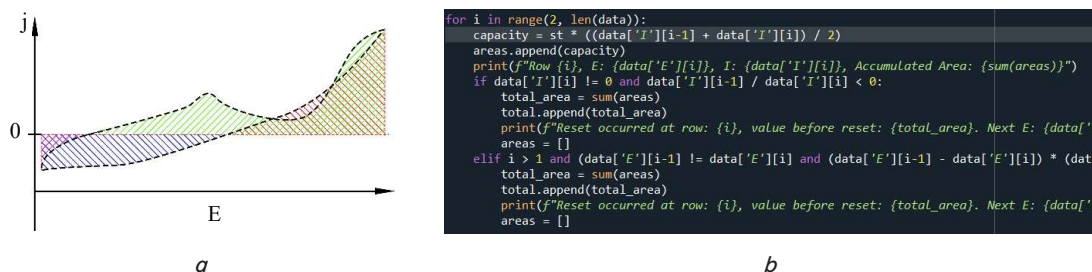


```python
for i in range(2, len(data)):
    capacity = st * ((data['I'][i-1] + data['I'][i]) / 2)
    areas.append(capacity)
    print(f"Row {i}, E: {data['E'][i]}, I: {data['I'][i]}, Accumulated Area: {sum(areas)}")
    if data['I'][i] != 0 and data['I'][i-1] / data['I'][i] < 0:
        total_area = sum(areas)
        total.append(total_area)
        print(f"Reset occurred at row: {i}, value before reset: {total_area}. Next E: {data['E'
        areas = []
    elif i > 1 and (data['E'][i-1] != data['E'][i] and (data['E'][i-1] - data['E'][i]) * (data
        total_area = sum(areas)
        total.append(total_area)
        print(f"Reset occurred at row: {i}, value before reset: {total_area}. Next E: {data['E'
        areas = []
```

a   *b*

Fig. 2. Calculation of charge based on cyclic voltammetry results: *a* — example of a cyclic voltammetry curve with marked zones for charge calculation; *b* — fragment of the code block for charge calculation in the program

5. Results of the study of heat-resistant superalloy in turbine elements

5. 1. Determination of the composition of the studied alloy

The composition of the superalloy, calculated from several measurements, is presented in Table 1.

Table 1

Comparison of the composition of the unknown alloy with the JS32-VI superalloy

Composition of elements by mass for JS32-VI, %											
Ni	Cr	Co	W	Al	Re	Ta	Nb	C	Mo	B	Ce
60.9	4.9	9.0	8.5	5.9	4.0	4.0	1.6	0.15	1.0	0.02	0.02
Composition of elements by mass for the unknown superalloy, %											
Ni	Cr	Co	W	Al	Re	Ta	Nb	C	Mo	B	Ce
60.7	4.5	8.9	8.22	5.8	3.5	3.5	1.6	0	1.2	0	0

The analysis of the composition of this superalloy allows conclusions to be drawn regarding its purpose and possible grade. It is evident that this is a nickel-based superalloy (60 % Ni), and the alloy also contains rhenium and other rare or refractory metals (Co, Nb, Mo, Ta, W). In addition to Cr and Al, the other elements in the superalloy are present in relatively small amounts.

5. 2. Determination of the reactivity of the alloy during anodic dissolution in selected electrolyte solutions

Cyclic voltammetry (CV) was used to determine and compare the electrochemical properties. For comparison, it was decided to also record CV curves for pure nickel under similar conditions, as nickel is the primary component of the alloy.

Fig. 3 shows the CV curves for nickel and the superalloy in a sodium chloride solution. A comparison of the two CVs reveals a significant difference in the electrochemical behavior of the materials. For nickel, an oxidation peak is observed on the reverse potential scan, and the cathodic current densities do not exceed 5 mA·cm^{-2}. In contrast, the superalloy shows no peaks on either the forward or reverse potential scans, and there is significant hysteresis.

In the solution of methanesulfonic acid used as the electrolyte, nickel also exhibits two peaks, but only during the forward potential scan (from lower to higher values), with no peaks present on the reverse scan (Fig. 4). The current densities are much lower compared to those obtained in the chloride solution. For the superalloy in the CH_3SO_3H solution, the CV curve shape is similar to that recorded in NaCl, but the current densities are also significantly lower.

An interesting distinction is observed in the CV curves for the hydrochloric acid solution, Fig. 5. The CV curve for nickel shows almost no hysteresis, and all cycles are similar (Fig. 5, *a*). In contrast, the superalloy curve (Fig. 5, *b*) contains two peaks. Moreover, the second of the two peaks, located around +0.3 V and +0.8 V, appears on the reverse scan. This peak shifts its maximum with each cycle. It can be assumed that the absence of a constant current density may be related to changes in the concentration of ions that are further oxidized on the reverse scan. These ions could be metal ions that form salts with different oxidation states. The change in ion concentration could be due to their instability while in the electrolyte. The other peak, present during the forward scan, is stable and does not change over time.
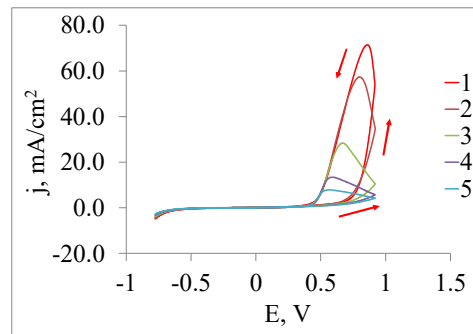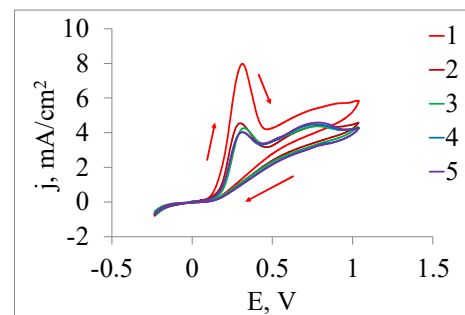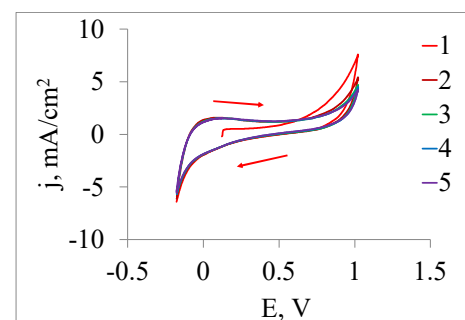

a
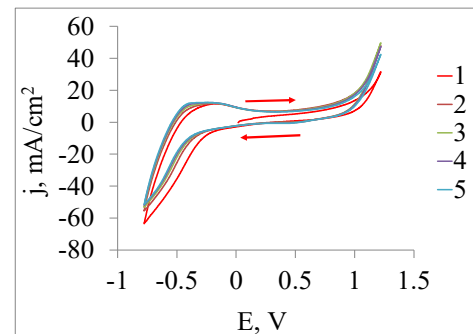
b

Fig. 3. Cyclic voltammetry curves obtained in 0.5 M NaCl solution, where the current density *j*, A·cm^{-2}, and potential *E*, V: *a* — Ni; *b* — Superalloy

a

b

Fig. 4. Cyclic voltammetry curves obtained in 0.5 M CH_3SO_3H solution, where the current density *j*, A·cm^{-2} and potential *E*, V: *a* — Ni; *b* — Superalloy

The last two curves for the sulfuric acid solution, in terms of behavior and shape, are similar to the CV curves for the methanesulfonic acid solution but with lower current densities, Fig. 7.
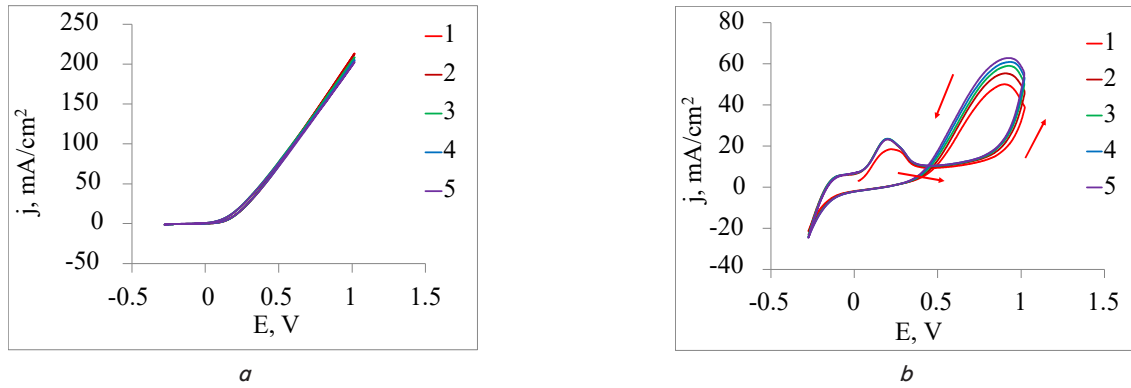
Fig. 5. Cyclic voltammetry curves obtained in 0.5 M HCl solution, where the current density j, A·cm^{-2}, and potential E, V: a — Ni; b — Superalloy
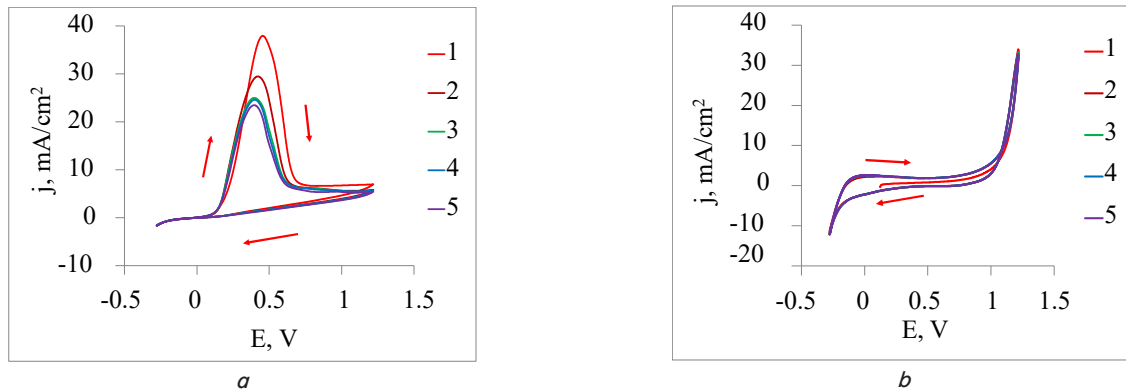

Fig. 6. Cyclic voltammetry curves obtained in 0.5 M H$_2$SO$_4$ solution, where the current density j, A·cm^{-2}, and potential E, V: a — Ni; b — Superalloy

Thus, it can be preliminarily concluded that the electrochemical behavior of the superalloy depends on the chosen electrolyte and is entirely different compared to pure nickel.

To evaluate the specific charge, integration of the CV was performed during the first cycle. Fig. 7 shows the obtained charge values in various electrolytes during the anodic dissolution of the superalloy and nickel.

The comparison of the obtained values allows conclusions to be drawn about the efficiency of the superalloy dissolution during the anodic process in specific electrolytes.
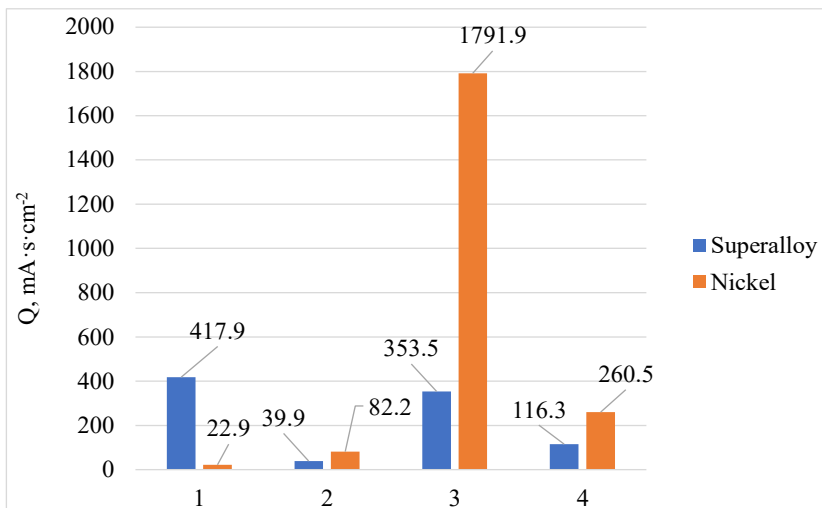

Fig. 7. Specific Charge Q, mA·s·cm^{-2}, determined for nickel and the investigated superalloy in various solutions: 1 — NaCl; 2 — CH$_3$SO$_3$H; 3 — HCl; 4 — H$_2$SO$_4$

6. Discussion of the results for the processing conditions of the heat-resistant superalloy

the analysis and comparison of the composition of the turbine elements' superalloy revealed that the turbine components from the destroyed military equipment are composed of a single alloy grade. A comparison of the unknown superalloy composition showed that its closest match among existing alloys is the JS32-VI superalloy (Table 1). The composition of JS32-VI is very close in percentage to the investigated superalloy [25]. The minor differences in percentages are likely due to local deviations, and element redistribution during operation in the turbine material under aggressive conditions, i.e., during fluctuating thermal and mechanical loads. Therefore, the investigated superalloy corresponds to the composition of the nickel-based JS32-VI superalloy, designed for high-temperature environments. A brief description of the JS32-VI superalloy is as follows: the JS32-VI alloy is a three-phase system consisting of a γ-solid solution based on nickel with an FCC crystal structure, dispersed precipitates of the strengthening γ'-phase based on the Ni$_3$Al intermetallic compound with an L1$_2$ crystal structure, and MC-type monocarbide based on Ta(Nb, W)C. The presence of some additional elements compared to the JS32-VI composition

may indicate contamination from fuel that is present on the surface and near-surface layers [25].

It should be noted that after the experiments, a black metallic deposit was found on the surface of the superalloy. During the experiments, the color of the solution gradually changed from clear to light green, corresponding to the color of nickel aquacomplexes. Since cathodic current can lead to the deposition of one or more dissolved metals during the anodic process, it can be assumed that one of the deposited metals in the residue is nickel, which constitutes more than 60 % of the alloy.

The CV curves for nickel and the superalloy were significantly different. Thus, although nickel is the main component of the superalloy (61 % by mass), their electrochemical activity varies sharply.

A comparison and analysis of the CV curves recorded in NaCl revealed significant differences. For nickel, a distinct peak is observed on the reverse potential scan, which gradually decreases (Fig. 3). This behavior may be due to insufficient Cl⁻ ion concentration in the electrolyte, and at relatively high potential sweep rates, the metal surface may partially passivate [26]. For the superalloy in this solution, no peaks are visible, and the CV curve shape remains relatively constant.

The CV curves for the methanesulfonic acid solution also differ sharply from those obtained in other solutions (Fig. 4). For nickel, two peaks are observed during the forward scan. The first peak is likely associated with nickel dissolution, which typically occurs in the potential range from −0.1 V to +0.3 V. The shape of the CV curve for the superalloy is similar to that of the superalloy in NaCl. It is worth noting that the current densities for the CV in the methanesulfonic electrolyte are the lowest among all the CVs.

The CV curves for the hydrochloric acid solution also differ significantly (Fig. 5). For nickel, the CV remains unchanged, with the highest current densities. This behavior is due to the use of a solution where dissolution occurs not only electrochemically but also chemically, making passivation less likely. The superalloy also has an unusual CV shape. A peak appears at around +0.2 V during the forward scan and another at around +0.7 V on the reverse scan. This is likely due to the oxidation of dissolution products formed during the forward potential scan.

Nickel in the sulfuric acid solution also exhibits a single peak during the forward scan, which is likely associated with the electrochemical oxidation of nickel (Fig. 6). In turn, the CV curve for the superalloy has no peaks and resembles the CV curves in CH_3SO_3H and NaCl.

To determine the dissolution efficiency, the specific charges for the anodic process were calculated for the first full cycle of the CV, as shown in Fig. 7. During the calculation, the portion of the CV from the current density equal to 0 mA/cm² to the positive value where the potential changes direction was used. This is equivalent to a single polarization curve and can therefore be used for this evaluation. The analysis of the charge values for different solutions revealed the following.

The worst dissolution results were observed in the methanesulfonic acid solution for both nickel and the superalloy, as the charge values were the lowest among all solutions. The best dissolution results for the superalloy were in HCl and NaCl, as these solutions produced the highest charges for superalloy dissolution (Fig. 7). This can be explained by the fact that both solutions contain Cl⁻ ions, which are powerful depassivators. Therefore, the most promising solutions for the dissolution and subsequent processing of the superalloy are NaCl and HCl solutions.

The limitation of this study is that the results may apply only to superalloys with similar structures and compositions, such as JS26U, JS30, JS28, JS36, and JS40.

The next step in the research could involve testing the electrochemical dissolution of alloys in NaCl and HCl solutions with varying concentrations and current densities to identify optimal parameters. It is also important to conduct electrochemical dissolution using optimized parameters for alloy fragments in rotating titanium baskets. This would help increase dissolution speed by renewing the anodic material surface and partially reflecting fragments, thus increasing the dissolution surface area.

7. Conclusions

1. The composition of the unknown superalloy has been determined, and it corresponds to the composition of JS32-VI.

2. It has been determined that the most promising solutions for electrochemical dissolution and subsequent processing of the superalloy are those containing chloride ions. According to the data obtained, the amount of charge used for superalloy dissolution in 0.5M NaCl and HCl was 417.9 and 353.5 mA·s·cm⁻², respectively.

Conflict of interest

The authors declare no conflicts of interest regarding this research, including financial, personal, authorship, or any other factors that could influence the research and its results presented in this article.

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Data availability

The manuscript has no associated data.

Use of artificial intelligence

The authors confirm that no artificial intelligence technologies were used in the creation of this work.

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