W-based superalloys are widely used as elements of drilling equipment, high-speed steel cutting tools, or penetrators for armor-piercing munitions. Used or broken superalloy products are valuable waste that can be recycled to recover valuable components. The most economically and technologically viable method for recycling superalloy scrap is a selective treatment with the dissolution of the binder metal and the production of non-oxidized tungsten powder. The aim of this work was to determine the possibility of anodic treatment of the VNZh90 superalloy scrap with the selective dissolution of the binder metal. The anodic behavior of the VNZh90 superalloy (5 % Ni, 5 % Fe, 90 % W) in HCl solutions with a concentration (wt %) of 9, 13, 17, and 30 was studied by voltammetry. It was shown that the anodic polarization curves of the alloy contained two dissolution peaks on a fresh surface (Fe and Ni components of the binder metal) with a further decrease in the current density. The effect of significant passivation of the VNZh90 alloy was revealed: repeated polarization curves in a 9 % HCL solution contained only the Ni dissolution peak with a 6-fold reduced current density. The passivation of the VNZh90 alloy was explained by the depletion of the surface due to the dissolution of the active Fe component and the Ni passivation due to the W dissolution during the formation of a superalloy. An increase in the HCl concentration did not reveal an activating effect. It was found that there was no activation effect when $FeCl_3$ was added to the electrolyte. The introduction of NaCl showed a high activation effect, and the dissolution current density of the passivated Ni component increased by 1.69 times. The efficiency of selective dissolution of the binder metal of the highly passive VNZh90 alloy must be confirmed by the galvanostatic or volt-static method

Keywords: superalloy, passivation, anodic behavior, tungsten, nickel, iron, selective anodic dissolution

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INVESTIGATION OF THE ANODIC BEHAVIOR OF W-BASED SUPERALLOY FOR ELECTROCHEMICAL SELECTIVE TREATMENT

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1. Introduction

Superalloys are pseudo-alloys consisting of W or WC powders, or other hard components and a binder metal Ni, Co, Fe), obtained by powder metallurgy methods [1, 2]. Carbide alloys have high strength and heat resistance, which allow them to be widely used in various fields: as a material for geological drill bits, high-speed cutters for processing steel, armor-piercing penetrators, rocket nozzles. Brittleness and low plasticity [3] make them vulnerable to shock loads and deformations. However, the high cost of raw materials and the monopoly on them in some countries make the idea of recycling different types of superalloys, for example, for cutting tools production, increasingly attractive [4]. Recycling can be carried out in various ways, for example, by the hydrometallurgical method [5]. However, superalloys have increased corrosion resistance, which complicates hydrometallurgical reusing [6]. It is proposed to use other methods that make it possible not only to recycle materials to obtain superalloys [7] but also to use the extracted components in other areas – for example, to obtain $Ni(OH)_2$ as an active substance of supercapacitors [8] and alkaline batteries [9, 10]. Recycling of binder metal is also proposed [11, 12], in particular Ni [13] and Co [14]. Obtaining ultrafine (10–50 μm) binder metal powders, and especially solid components, in particular W, is a complex and expensive process. Therefore, the development of simple methods for obtaining tungsten powder from superalloy scrap, as the most valuable and expensive component, is very urgent. However, the development of such methods is impossible without studying the characteristics of specific superalloys.

2. Literature review and problem statement

Recycling or reusing of any materials is primarily determined by their structure. Superalloys, also called pseudo-alloys, are not true alloys and are composite materials.

Usually, composite materials, both organic [15] and inorganic [16], consist of a matrix former and a filler material. At the same time, superalloys have a different structure: a large number of particles of hard material are held together by a molten binder metal (mainly Ni or Co). Due to the high hardness and method of production, mechanical recycling of superalloy scrap is practically impossible. For the recycling of superalloy scrap, complete dissolution of all components of the superalloy or selective treatment can be used.

Complete dissolution can be carried out by a chemical method, in particular, by treatment with hot sulfuric acid [7], hydrochloric acid [7, 17], hydrochloric acid with a purge of gaseous chlorine [7, 13], or thermal reduction with carbon [7]. Pyrometallurgical methods are also used [7]. For complete dissolution, electrochemical methods are used, in particular, anodic dissolution in a sodium chloride solution [4], or in an acidic solution [7]. In [18], an electrochemical method was applied. However, the method was used not for the direct anodic dissolution of superalloy scrap but for the production of gaseous chlorine, which oxidized the superalloy surface prone to passivation.

The main disadvantage of complete dissolution is that for the complete regeneration of the hard component, especially tungsten or tungsten carbide, it is necessary to obtain ultrafine tungsten oxide and reduce it with hydrogen, which significantly increases the cost of the secondary hard component.

The alternative is the selective dissolution of the binder metal. Methods of selective treatment in melts of a mixture of metals [5] as well as in molten zinc [19] have been proposed. During treatment, the binder metal is dissolved, and then the metal-solvent is distilled off under a vacuum. However, this method has a significant drawback – impurities of zinc and other more volatile metals remain in the regenerated nickel or cobalt. As a result, during the manufacture of superalloy products, these impurities evaporate, forming cavities in the product, and the mechanical properties of the product are sharply reduced. This method is not applied for the recycling of components for the manufacture of superalloys.

Selective dissolution of the binder metal can be carried out electrochemically. Electrochemical dissolution of superalloy scrap in acidic solutions was proposed [17], including at a controlled potential [20]. However, a detailed analysis of publications shows that the process of anodic dissolution is proposed to be carried out at such current densities at which the anodic potential of the superalloy reaches values of 1500–2000 mV. Under these conditions, tungsten and tungsten carbide will oxidize, precipitating in the oxidized form – tungsten oxide or tungstic acid. The method is selective since the binder metal goes into solution and the oxidized form of the hard component precipitates. The hard component is oxidized, and therefore, electrochemical anodic dissolution under the given conditions has the same disadvantages as complete dissolution that is the complexity of further recycling to obtain ultrafine tungsten or tungsten carbide.

Tungsten-containing superalloys can be basically divided into two groups. The first group includes alloys the hard component of which is tungsten carbide WC, for example, alloys of the VN or VK type. In the alloys of the second group, the hard component is particles of metallic tungsten (W). These are the VNZh type alloys. In [21], a method for the selective electrochemical recycling of superalloy scrap was proposed, both tungsten carbide (VN8, VK20KS) and tungsten (VNZh, VNDS). The recycling was carried out in an acidic solution. The high efficiency of selective anodic dissolution of the binder metal for WC-containing superalloys was shown. At the same time, it was shown that the VNZh90 superalloy had the lowest dissolution currents of the binder metal, which indicated high passivation of this alloy. To overcome passivity, a group of authors [22] proposed electrochemical anodic dissolution not in an acidic but an alkaline ammonium electrolyte. At the same time, to overcome passivity, it was proposed to carry out treatment with both direct [23] and alternating current [24]. The passivity of the VNZh type alloy was overcome. However, the use of an alkaline ammonium electrolyte indicated the possible dissolution of the hard component (W) with the formation of ammonium paratungstate. In this case, electrochemical recycling in an alkaline-ammonium electrolyte cannot be correctly called selective with the production of unoxidized tungsten powder.

At the same time, selective electrochemical treatment of the VNZh type superalloy scrap with dissolving only the binder metal is very promising since it occurs at a low temperature and allows obtaining tungsten powder as a hard component, which can be directly used for the manufacture of superalloys. However, to develop such a selective dissolution of the VNZh type alloy with the oxidation of only the binder metal, it is necessary to study the anodic behavior of this alloy in an acidic electrolyte.

3. The aim and objectives of the study

The aim of the work is to determine the possibility of anodic recycling of the VNZh type superalloy scrap with the selective dissolution of the binder metal.

To achieve the aim, the following objectives were set:

– to study the anodic behavior of the VNZh90 alloy in HCl solutions of various concentrations;

– to study the anodic behavior of the VNZh90 alloy in HCl solutions in the presence of activators.

4. Materials and methods for studying the anodic characteristics of the VNZh90 superalloy

Description of the investigated superalloy sample.

The VNZh90 alloy of the following composition was used for the study: 90 % W (hard component), 5 % Ni, 5 % Fe (binder metal). The investigated sample of the VNZh90 alloy was the penetrator for armor-piercing munitions.

Electrolyte composition for studying the anodic characteristics of the superalloy.

The choice of the electrolyte composition was based on the chemical properties of the hard component (tungsten) and the binder metal (nickel, iron) as well as on the possibility of passivation of these components. Based on the previous studies [21], 9 % (wt.) HCl solution was chosen as the base electrolyte. To study the effect of the concentration of hydrochloric acid, it was proposed to use the following series of concentrations: 13 %, 17 %, 30 %. To evaluate the activating effect, the following additives were introduced into the base electrolyte: NaCl (saturated solution) and $FeCl_3$ (10 g/L).

Method for studying the anodic characteristics of the superalloy.

To determine the possibility of selective dissolution of the binder metal, the anodic behavior of the samples of superalloys in the selected electrolyte was studied. Anodic polarization characteristics were measured using a specially designed cell. The working electrode was a VNZh90 superalloy sample. To supply current, a copper wire was wound on the sample and placed in specially prepared tubular PVC containers for isolation from the electrolyte. Mesh platinum was used as a counter electrode. Reference electrode – Ag/AgCl (sat.). Polarization curves were recorded in a potentiodynamic mode using an Ellins P–8 potentiostat (Russia) with an anodic scan from a stationary potential to a potential of +2000 mV with a scan rate of 5 mV/s. To evaluate the possibility of passivation, the anodic polarization curves were recorded twice. When replacing the electrolyte, the surface of the superalloy was refreshed.

5. Results of studying the anodic characteristics of the superalloy

Results of studying the anodic behavior of the VNZh90 alloy in hydrochloric acid solutions of various concentrations.

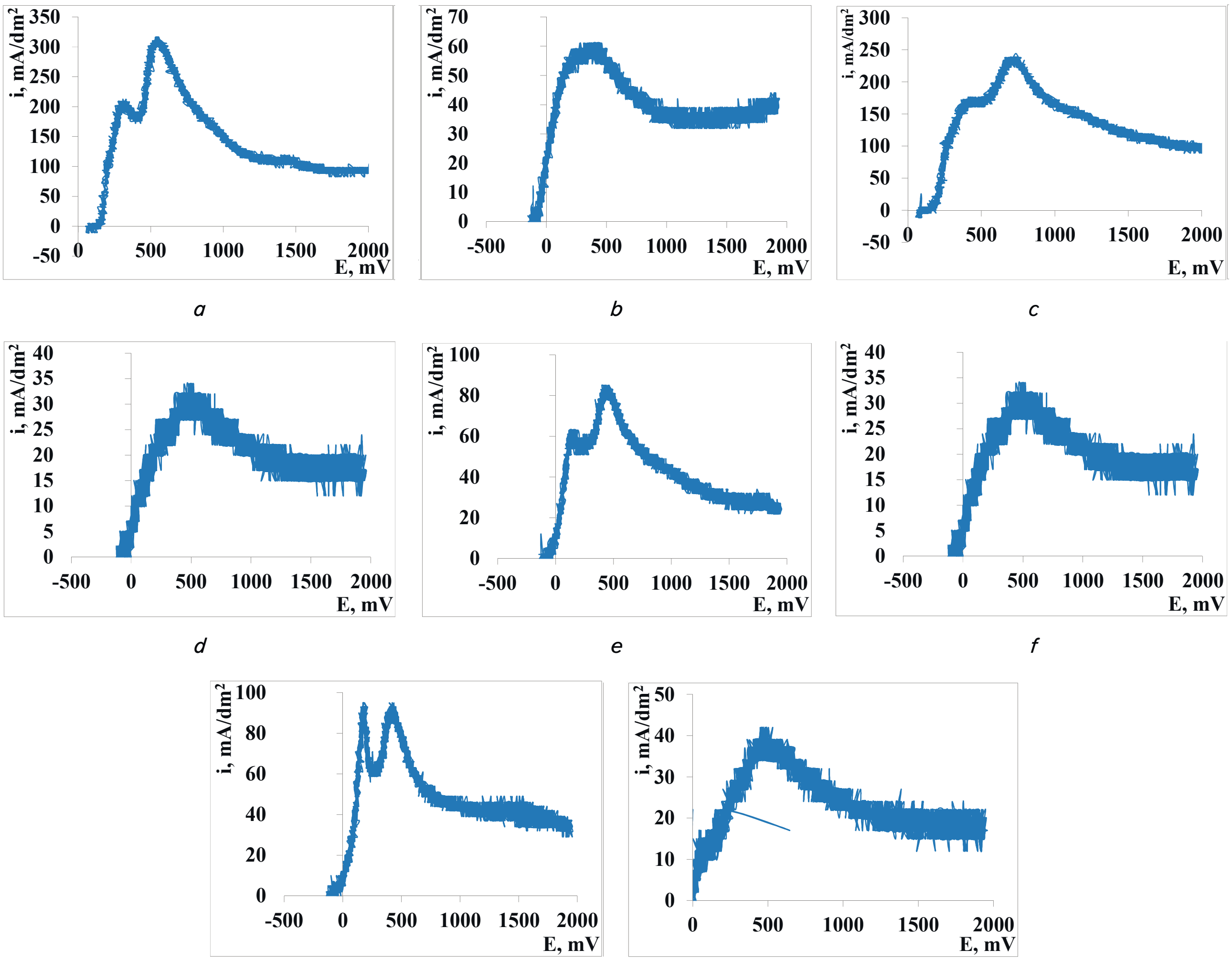
Anodic polarization curves are shown in Fig. 1. At all concentrations of HCl, the polarization curves had a similar character to the classical curve with passivation. On the first polarization curve (fresh alloy surface), there were two dissolution peaks with a further decrease in the dissolution current and the passivation plateau. The repeated polarization curve had only one dissolution peak.

In this case, the current of the dissolution peak and the current of the passivation plateau were significantly reduced, which indicated the passivation of the VNZh alloy. With an increase in the concentration of hydrochloric acid from 9 % to 17 %, the currents of the dissolution peaks and the currents of the passivation plateau decreased, and at a concentration of 30 %, they slightly increased.

Results of studying the anodic behavior of the VNZh90 alloy in hydrochloric acid solutions in the presence of activators.

Anodic polarization curves in the presence of activators (NaCl and $FeCl_3$) are shown in Fig. 2.

Anodic polarization curves recorded in the presence of activators also had the character of curves with passivation, which had a clearly pronounced one peak of anodic dissolution. With increasing potential, a passivation plateau was observed. In this case, the anode curve of the VNZh90 alloy, recorded in the presence of NaCl, was characterized by higher currents as compared to the curve recorded in the presence of $FeCl_3$.

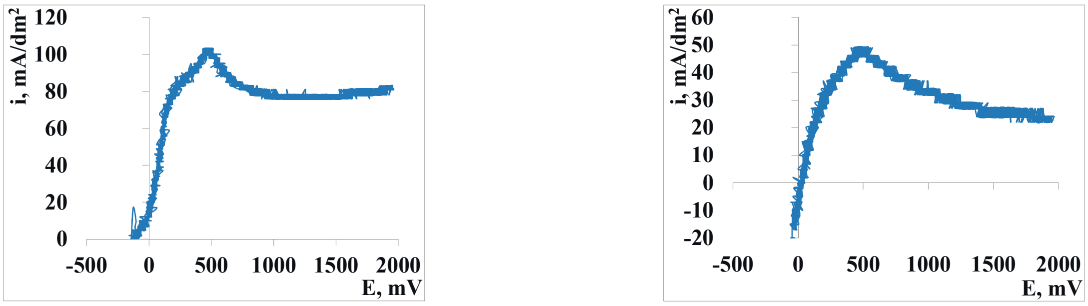


a *b* *c*

d *e* *f*

g *h*

Fig. 1. Anodic polarization curves of the VNZh90 alloy in HCl solution of various concentrations: *a*, *b* – 9 %; *c*, *d* – 13 %; *e*, *f* – 17 %; *g*, *h* – 30 %; *a*, *c*, *e*, *g* – the first polarization curve (fresh alloy surface); *b*, *d*, *f*, *h* – repeated polarization curve (without refreshing of the alloy surface)



a *b*

Fig. 2. Anodic polarization curves of the VNZh90 alloy in a 9 % HCl solution with anodic activators (without refreshing of the alloy surface): *a* – NaCl; *b* – $FeCl_3$

6. Discussion of the results of studying the anodic characteristics of the VNZh90 superalloy

Influence of the concentration of the hydrochloric acid solution on the anodic behavior of the VNZh90 alloy.

It was revealed that the polarization curves had a shape characteristic of the curve of passivated materials, which corresponded to the literature data [19, 20]. The results of the analysis of the digital characteristics of the polarization curves are given in Table 1.

Table 1

Electrochemical characteristics of the anodic polarization curves of the VNZh90 alloy in HCl solutions of various concentrations

Dissolution peak		Passivation plateau		Dissolution peak		Passivation plateau	
E, mV	i, mA/dm^2	E, mV	i, mA/dm^2	E, mV	i, mA/dm^2	E, mV	i mA/dm^2
9 % first curve				9 % repeated curve			
300	198	Not clearly expressed	93 (at E=2,000 mV)	330	61	870–1,475	39–32
535	306						
13 % first curve				13 % repeated curve			
400	161	Not clearly expressed	98 (at E=2,000 mV)	460	51	1,220–2,000	27–22
680	219						
17 % first curve				17 % repeated curve			
140	56	Not clearly expressed	24 (at E=2,000 mV)	475	29	1,265–2,000	20–15
430	78						
30 % first curve				30 % repeated curve			
185	93	785–2,000	49-34	450	37	1,190–2,000	22–17
415	93						

It was found that for all HCl concentrations, the anodic polarization curves for the fresh alloy surface and the repeated polarization curves were fundamentally different. The first polarization curves (Fig. 1, *a*, *c*, *e*, *g*) had two peaks of anodic dissolution, after which the dissolution current density dropped, and constantly decreased to a potential of +2,000 mV. The first peak corresponded to the dissolution of iron; the second peak corresponded to the dissolution of nickel. A sharp decrease in the dissolution current density clearly indicated significant passivation of the alloy. On the repeated polarization curves, only one dissolution peak and a well-defined passivation plateau were observed. It should be noted that the potentials of the dissolution peaks indicated the dissolution of Ni, while the peak of Fe dissolution was not observed. Comparison of the dissolution peak currents for the first and repeated polarization curves in a 9 % HCl solution showed a decrease from 306 mA/dm^2 to 61 mA/m^2, i.e. 6 times. The current of the passivation plateau of the repeated polarization curve was almost 3 times lower than the dissolution current at a final potential of 2,000 mV on the first polarization curve. All these data indicated a very significant passivation of the VNZh90 alloy. The work [21] showed the fundamental differences between superalloys with a WC-hard component and superalloys with a W-hard component. Both types of superalloys were produced by powder metallurgy when a mixture of ultrafine powders of a hard component and a binder metal was heated under pressure to semi-melt the binder metal. In the case of the tungsten carbide hard component, the dissolution of WC in the binder metal did not occur. At the same time, in the case of a tungsten superalloy, W was able to dissolve in nickel, which led to significant passivation of nickel. In this case, tungsten did not dissolve in Fe. As a result, when the first polarization curve was recorded, dissolution peaks of nickel and iron were observed. As a result, the surface of the VNZh90 superalloy was depleted in the active Fe-phase, and the proportion of W of the hard component and passivated Ni increased. As a result, only the Ni dissolution peak was present on the repeated polarization curve, and the currents were greatly reduced. The strong oscillation of the values of currents and potentials on the repeated polarization curve confirmed the unevenness of the surface of the VNZh90 alloy. The phenomenon of significant passivation of the surface of the VNZh90 alloy actually prevented the selective dissolution of the binder metal. Initially, based on the known tendency of Ni to passivation, a hydrochloric acid solution was chosen as the electrolyte, based on the strong depassivating effect of the chloride ion. For possible depassivation of the Ni-bond in the VNZh90 alloy, the HCl concentration was increased. It should be noted that an increase in the concentration of hydrochloric acid can have a double effect: the effect of increased acidity (concentration of H^+) and the effect of an increased concentration of a depassivator. An increase in concentration from 9 % to 17 % led to an increase in the dissolution currents of nickel and iron in the first polarization curve, which indicated an increased dissolution of the active components of the binder metal. However, on the repeated polarization curve, the current of the dissolution peak of the passivated Ni component of the binder metal decreased from 61 mA/dm^2 (9 % HCl) to 51 mA/dm^2 (17 % HCl). With an increase in the concentration of hydrochloric acid in the series "9 % – 13 % – 17 % – 30 %", the series of currents of the dissolution peak of the passivated Ni component on the repeated polarization curve was "61 mA/dm^2 – 51 mA/dm^2 – 29 mA/dm^2 – 37 mA/dm^2". In this case, with increasing concentration, the current of the dissolution peak on the repeated curve decreased, and only at 30 % concentration, the peak current slightly increased. Thereby, it should be noted that an increase in the HCl concentration resulted in rapid dissolution of the active components of the binder metal and did not lead to anodic activation of the passivated component (Ni with dissolved W). However, the use of concentrated hydrochloric acid (30 %) gave a slight activating effect.

Anodic behavior of the VNZh90 alloy in 9 % hydrochloric acid solutions in the presence of activators.

It was proposed to use NaCl and $FeCl_3$ as activators for the anodic dissolution of the binder metal of the VNZh90 alloy. The use of NaCl was based on the introduction of a depassivator (Cl^-) while maintaining the electrolyte acidity level. The use of $FeCl_3$ as an activator was based both on the introduction of an additional amount of chloride ions as depassivators and on the high oxidation potential of the Fe^{3+}/Fe^{2+} redox pair. These characteristics allowed the use of $FeCl_3$ for etching copper circuit boards.

The results of the analysis of the digital characteristics of the repeated polarization curves recorded in the presence of activators are shown in Table 2.

Table 2

Electrochemical characteristics of the repeated anodic polarization curves of the VNZh90 alloy in 9 % HCl solutions in the presence of activators

Dissolution peak		Passivation plateau		Dissolution peak		Passivation plateau	
E, mV	*i*, mA/dm^2	E, mV	*i*, mA/dm^2	E, mV	*i*, mA/dm^2	E, mV	*i*, mA/dm^2
NaCl activator				$FeCl_3$ activator			
220	83	940–1,580	76	450	44	1,400–2,000	27
430	103						

Comparative analysis of the data of the polarization curves in the presence of activators (Table 2) with the parameters of the repeated polarization curve in 9 % HCl solution (Table 1) showed that the introduction of sodium chloride (as an activator) led to the appearance of two dissolution peaks on the repeated polarization curve corresponding to the Fe dissolution and passivated Ni components of the binder metal. The dissolution peak of the Fe component was absent on the repeated polarization curve in a 9 % HCl solution without an activator. In this case, the peak current density of the Ni dissolution in the presence of a NaCl activator was 1.69 times higher than in an electrolyte without an activator (103 mA/dm^2 and 61 mA/dm^2, respectively). This led us to two conclusions. First, it was necessary to point out the high activating ability of NaCl concerning the anodic dissolution of the VNZh90 alloy. Second, the appearance of the Fe dissolution peak indicated the overcoming of the surface passivation, and probably indicated that the passivation of the nickel component occurred through the surface dissolution of tungsten nickel grains by tungsten. When this layer of the solid solution of tungsten in nickel was depassivated, the Ni dissolution was accelerated. The introduction of $FeCl_3$ into the hydrochloric acid solution did not give an activating effect: only the Ni dissolution peak with a current density of 44 mA/dm^2 appeared on the anodic curve, while the current of the nickel dissolution peak on the repeated polarization curve in the hydrochloric acid solution without activator was 61 mA/dm^2.

To sum up, it should be noted that selective anodic dissolution of the binder metal of the VNZh90 alloy is possible in a NaCl solution with 9 % (wt.) HCl. The limitation of the study is that the efficiency of the selective dissolution of the binder metal of the highly passive VNZh90 superalloy is shown only by the method of studying the anodic behavior of the sample. It is necessary to confirm these data by the galvanostatic or volt-static methods. It is also advisable to further search for additional activators.

7. Conclusions

1. The anodic behavior of the VNZh90 superalloy with a W hard component in HCl solutions with mass concentrations of 9 %, 13 %, 17 %, and 30 % has been studied. The anodic polarization curves of the alloy with a fresh surface contain the dissolution peaks of the Fe and Ni components of the binder metal with a further decrease in the current density. The repeated polarization curves contain only the Ni dissolution peak and the passivation plateau. In this case, in a 9 % NaCl solution, the current density of the Ni dissolution peak has decreased 6 times. The disappearance of the Fe dissolution peak and a sharp decrease in the current density of the Ni peak are explained by the depletion of the VNZh90 alloy surface due to the dissolution of the active Fe component and the Ni passivation as a result of the W dissolution during the formation of a superalloy. An increase in the concentration of hydrochloric acid leads to a decrease in the current density of the dissolution peak of the passivated Ni component, which indicates the absence of an activating effect.

2. The anodic behavior of the VNZh90 superalloy in a 9 % HCl solution in the presence of NaCl and $FeCl_3$ has been studied. The absence of the activating effect of $FeCl_3$ addition has been revealed. The introduction of NaCl has shown a high activating effect; the current density of dissolution of the Ni passivated component has increased by 1.69 times. An additional effect of the NaCl activation is the appearance of the Fe dissolution peak on the repeated anodic polarization curve. This has indicated that the Ni component is passivated due to the dissolution of the Ni particles in the surface layer. Depassivation of this layer has led to the more active Ni dissolution and an increase in the depth of the dissolution process of the binder metal. In this case, the possibility of selective anodic dissolution of the binder metal of the highly passive VNZh90 alloy in a NaCl solution with 9 wt % HCl has been shown. The effectiveness of the selective dissolution must be confirmed by galvanostatic or volt-static methods.

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