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ANODIC DISSOLUTION AND CORROSION OF Al IN KOH SOLUTIONS IN 90% ETHANOL CONTAINING ADDITIVES OF GALLIUM AND INDIUM COMPOUNDS. INHIBITORY EFFECT OF CITRIC ACID

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Abstract. The influence of addition of citric acid monohydrate on anodic dissolution and corrosion rate of aluminum in KOH solutions in 90% ethanol containing additives of gallium and indium compounds has been considered. It is shown that the introduction of citric acid monohydrate into the solution allows to reduce the magnitude of aluminum corrosion current without reducing the rate of its anodic dissolution. The inhibition efficiency of citric acid monohydrate when introduced into the solution at a concentration of $5 \cdot 10^{-4}$ M is 58%. The discharge galvanostatic curves in the above electrolyte show a discharge plateau up to a discharge current density of 16 mA/cm^2.

Keywords: *aluminum, KOH, ethanol, anodic dissolution, hydrogen release, corrosion current*

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INTRODUCTION

Aluminum, due to its high volumetric specific capacity (8.04 mA h/cm^3) and the value of the standard electrode potential (−1.66 V), is a promising anode material for creating energy-intensive current sources. These features of aluminum and its widespread occurrence in the Earth's crust have long attracted the attention of researchers working in the field of chemical power sources. An element with an aluminum anode in a solution of nitric acid as an electrolyte was proposed by Buff as early as 1897. The EMF of this current source is 1.377 V [1]. Later, various salt, acid, and alkaline water-based electrolytes were considered in the works on creating current sources with an aluminum anode [2–4].

A significant limitation of the use of aluminum as an anode in current sources with an aqueous electrolyte is due to the significant self-dissolution of aluminum caused by the active release of hydrogen. To overcome this complication, many researchers have switched to using non-aqueous electrolytes [5–9]. The electrochemical behavior of aluminum depends not only on the electrolyte used, but also on the pretreatment of its surface. The use of aluminum as an anode in non-aqueous electrolytes requires the use of labor-intensive pretreatment necessary to remove the oxide film on the electrode [7, 9]. This complicates the practical use of aluminum as an anode in current sources.

The use of alkaline water-alcohol solutions [10–16] makes it possible to overcome this complication, since the destruction of the insulating oxide layer on the electrode occurs directly upon contact of aluminum with the working electrolyte. The electrochemical activity of the aluminum electrode in these solutions depends on the water content in the electrolyte, increasing with increasing concentration [11, 12, 15]. Simultaneously with an increase in the activity of aluminum, an increase in the water content leads to an increase in the rate of its corrosion. A number of corrosion inhibitors used in aqueous solutions have also proved effective in aqueous alcohol electrolytes. In [13], the effect of Na_2SnO_3 on the anodic behavior of aluminum and the rate of its corrosion in a 4 M KOH solution in a mixed solvent methanol/water (volume ratio 4/1) was studied. The introduction of sodium stannate significantly slows down the corrosion of aluminum due to the deposition of tin on its surface. The authors of [13] found that with a significant increase in the concentration of sodium stannate in the electrolyte, the tin precipitate on the surface of the aluminum electrode cracks, leading to a decrease in the inhibitory effect of the stannate. The introduction of Na_2SnO_3 simultaneously improves the discharge characteristics of aluminum by inhibiting the formation of a dense deposit of reaction products on the electrode surface. This effect increases with increasing stannate concentration in the electrolyte. At a sodium stannate concentration of 10.0 mM/l, galvanostatic curves with a discharge current density up to 20 mA/cm^2 demonstrate a discharge plateau. At the same current density, the discharge curves in solutions containing stannate are shifted towards negative potential values compared to the curves obtained in solutions without an inhibitor. In [17], a hybrid inhibitor was proposed for aluminum-air current sources, including sodium stannate and casein. The introduction of 0.05 M Na_2SnO_3 and 0.6 g/l casein into a 4 M NaOH solution reduces the rate of aluminum corrosion by an order of magnitude and increases the discharge capacity of the current source by 89.3%. The presence of ethylene glycol in the hybrid Na_2SnO_3/ ethylene glycol inhibitor [18] promotes a more uniform and dense deposition of tin on the surface of the aluminum electrode, which makes it possible to enhance the inhibitory effect of sodium stannate. At the same time, the discharge characteristics of the anode are improved. The best results are achieved by using 10% ethylene glycol (mass fraction) and 0.05 M Na_2SnO_3. In [18], a study of the corrosion behavior of the 1060 Al alloy in a 4 M aqueous NaOH solution showed that the use of the above hybrid inhibitor makes it possible to increase the use of the anode from 16% to 43%, and the specific energy density from 543 to 1577 Wh/kg. Zinc oxide is known to be an effective inhibitor of aluminum corrosion in alkaline electrolytes. The effectiveness of aluminum corrosion inhibition in KOH solutions depends on both the concentration of ZnO and the concentration of KOH [19]. The authors of [19] established that in KOH solutions saturated with zinc oxide, a dense zinc film forms on the aluminum surface, which has strong adhesion and maximum protective properties. The open-circuit potential of the aluminum electrode shifts towards positive values when ZnO is introduced. In a 55% KOH solution saturated with ZnO, this displacement is ~200 mV. With anodic polarization, the discrepancy between the polarization curves in a pure KOH solution and in the presence of ZnO additives decreases. The rate of aluminum corrosion in the above electrolyte decreases by 2 orders of magnitude compared to what is observed in the absence of ZnO additives.

A number of researchers have proposed hybrid inhibitors based on the combined use of zinc oxide and specially selected organic compounds. The effect of ZnO additives on the corrosion behavior of aluminum in a 4 KOH solution in a mixed methanol/water solvent (3/2 by volume ratio) was considered in [14]. The corrosion rate of aluminum in this solution (6.46 mA/cm^2) is significantly lower than in the corresponding 4 M aqueous KOH solution. The addition of ZnO to the electrolyte makes it possible to further reduce the rate of aluminum corrosion, and this effect can be enhanced by simultaneous administration of hydroxytryptamine into the solution. The introduction of 0.2 M ZnO and 1.0 ml/l hydroxytryptamine into the solution reduces the corrosion current of aluminum by 82 times, reducing it from 6.46 to 0.079 mA/cm^2. At the same time, the presence of zincate in the solution leads to a shift in the open-circuit potential of the aluminum electrode by 300 mV towards positive values. In [20], the effect of the combined use of zinc oxide and polyethylene glycol (**PEG**) on the electrochemical behavior and corrosion rate of aluminum in a 4 M KOH solution was considered. The authors [20] note that in the absence of PEG, the zinc precipitate on the aluminum surface has a loose, spongy structure and weak adhesion. In this case, it cannot be ensured that aluminum corrosion is inhibited for a long time. The introduction of PEG makes it possible to improve the characteristics of the zinc precipitate. The effectiveness of PEG depends on its concentration in the solution. The maximum effectiveness of this inhibitor is achieved by introducing 0.2 M ZnO and 2 mM PEG into the solution. The introduction of an inhibitor makes it possible to significantly reduce the rate of aluminum corrosion, at the same time, the conductivity of aluminum shifts by about +0.5 V. relative to that in the absence of an inhibitor. Accordingly, the discharge curves proceed at relatively low values of the electrode potential. The authors of [21] proposed a hybrid inhibitor including ZnO and α, ω-Bis (2-carboxymethyl) polyethylene glycol (PEG-diacid). The introduction of PEG-diacid affects the morphology of the zinc layer formed on the surface of aluminum, increasing its density and reducing porosity. The ZnO/PEG diacid inhibitor impedes the corrosion of aluminum in alkaline solutions by reducing the rate of the cathode reaction. Its use provides effective protection of the aluminum electrode from corrosion without affecting its discharge characteristics. During the discharge of the aluminum anode, the protective layer formed in the presence of an inhibitor is quickly removed from the surface of the aluminum anode, ensuring its effective discharge. Upon termination of the discharge, the protective layer of ZnO/PEG-diacid is restored, resuming protection of the anode from corrosion in the time intervals between discharges. The effectiveness of this inhibitor depends on both the amount of ZnO in the solution and the concentration of PEG-diacid. The optimal effect is achieved by introducing 5000 ppm PEG diacid and 16 g/l of ZnO into the solution.

The effect of calcium oxide and sodium and potassium citrate additives on the corrosion and anodic behavior of aluminum Al-2S in 4 M aqueous solutions of sodium and potassium hydroxide was studied in [22]. The authors of [22] suggest that aluminum in an alkaline

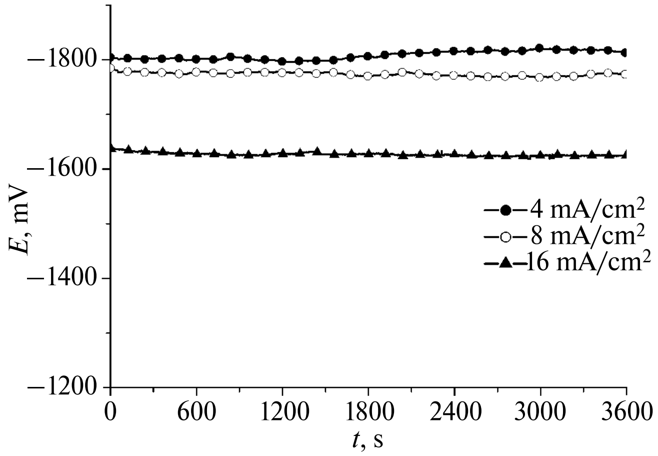

Fig. 1. Galvanostatic discharge curves on an Al electrode in a 2 M KOH solution in 90% ethanol containing 10^{-4} M Ga^{3+} and 10^{-3} M In^{3+} at various values of current density.

solution in the form of aluminate ions, interacting with Ca^{2+} ions, forms calcium aluminate, which precipitates as a thin film on the surface of aluminum, preventing its corrosion. At the same time, A1$^{3+\,\mathrm{i}}$ons, reacting with an excess of citrates, form aluminum citrate. Thus, calcium and aluminum citrates can also have an inhibitory effect on aluminum corrosion. It has been shown that the effectiveness of inhibition increases with increasing concentrations of both calcium oxide and sodium and potassium citrates. The inhibition efficiency in these solutions reaches 93%.

A study of the effect of calcium ions and tartrate ions on the behavior of aluminum in a 4 M aqueous KOH solution [23] showed that tartrate ions themselves have a slight inhibitory effect, but significantly enhance the inhibitory effect of calcium ions. The inhibition efficiency is 85%. The combined effect may be associated, according to the authors, with the formation of complexes leading to an increase in the solubility of $Ca(OH)^2$. In [24], the inhibitory effect of urea and thiourea on the corrosion behavior of the Al-Mg-In-Mn alloy in a 5 M aqueous KOH solution was investigated. It has been shown that the introduction of the above corrosion inhibitors into a solution in an amount of 25 mM makes it possible to reduce the corrosion current density of the alloy from 26 mA/cm^2 in a solution containing no inhibitors to 12.6 and 11.2 mA/cm^2 in the case of urea and thiourea additives, respectively. The effectiveness of the effect of urea and thiourea on the corrosion current grows with an increase in their concentration in solution.

Many of these inhibitors could not be used by us due to their low solubility in ethanol. Citric acid is one of the most effective corrosion inhibitors of aluminum and its alloys in an aqueous alkaline environment [25], however, it is highly soluble in both water and ethanol. These properties of citric acid led to its choice as an inhibitor in this work.

Earlier [15], we examined the effect of additives of gallium and indium compounds on the electrochemical activity of Al in a 2 KOH solution in 96% ethanol. It has been shown that the activating effect of gallium ions is most effective when their content in solution ranges from 10^{-5} to 10^{-4} M. Discharge galvanostatic curves in KOH solutions in 96% ethanol with additives of gallium and indium compounds at concentrations of 10^{-4} M and 10^{-3} M, respectively, make it possible to obtain a discharge plateau at current densities up to 4 mA/cm^2.

The purpose of this study is to search for the composition of an electrolyte based on a mixed aqueous-ethanol solvent that provides the maximum possible current of anodic dissolution of aluminum with a minimum current of its corrosion.

EXPERIMENTAL METHODS

Al electrodes were obtained from aluminum sheet of 99.999% purity. To carry out electrochemical measurements, smooth sheets 0.5 mm thick were cut into 5 x 10 mm rectangles with a tap for electrical contact. The surface of the Al electrode was treated with P 400 grit sandpaper and degreased with ethyl alcohol before measurement. All measurements were carried out in a 2 M KOH solution in 90% ethanol containing $Ga(NO_3)_3$ and $In(NO_3)_3$ additives. Electrolyte solutions were obtained using chemically pure reagents. 90% ethanol was used as a solvent.

The measurements were carried out in a small (15 cm^3) glass three-electrode cell. All measurements were carried out after preliminary exposure of the Al electrode in the working solution for 1 h. The reference electrode was a silver chloride electrode. The auxiliary electrode is made of platinum. Galvanostatic measurements at current densities of 4.8 and 16 mA/cm^2 were performed using an IPC2000 Pro potentiostat (EKONIKS, Russia). All measurements were performed at a temperature of 25 °C.

The traditional method of measuring the rate of gas release on aluminum in the working electrolyte was used. A small sample of the aluminum sheet used to make the electrodes was placed in a vessel with the test solution. The gas released on the aluminum sample was fed into an upside-down burette filled with water to measure the volume of released gas by the volume of displaced water. The volume of the released gas was brought to normal conditions by applying the ratio $V = V_m \cdot 273 \cdot (P - P_w)/(760 \cdot (273 + t))$. Here V is the volume of gas brought to normal conditions, V_m is the measured volume of the released gas, P is atmospheric pressure, P_w is the partial pressure of water vapor under experimental conditions,

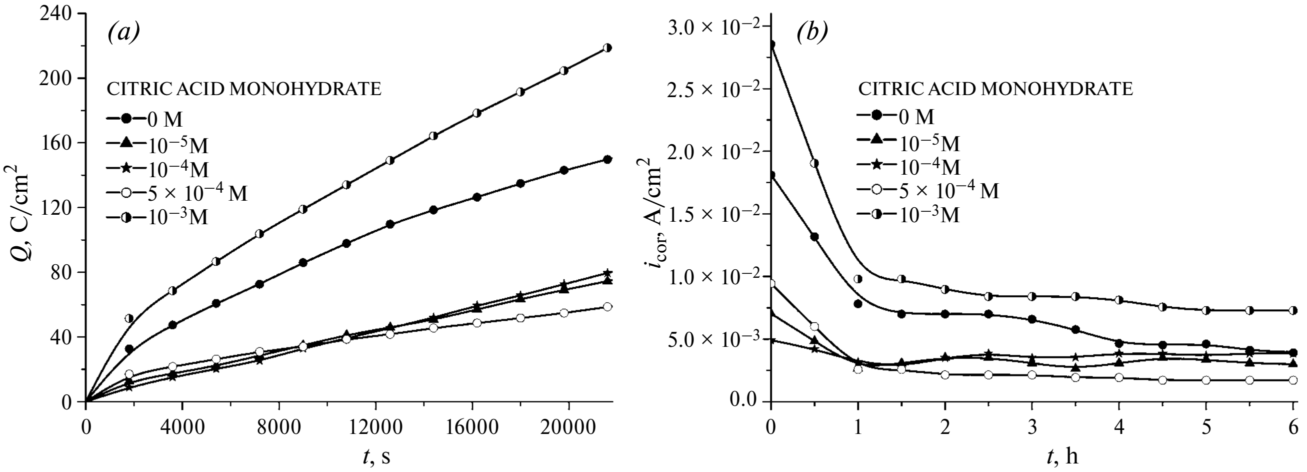

Fig. 2. Dependences: (a) of the amount of electricity spent on hydrogen release on Al, (b) of the current density of Al corrosion on the time it was in a 2 M KOH solution in 90% ethanol containing 10^{-4} M Ga^{3+} and 10^{-3} M In^{3+} and additives of citric acid monohydrate.

t is the temperature in °C. The amount of electricity (Q) spent on hydrogen reduction and the volume of hydrogen released, related to the surface unit of the aluminum sample under study, are related by the ratio $Q = 2FV/V_0$, where F is the Faraday number, V is the volume of released gas reduced to normal conditions, and V_0 is the volume of one mole of gas under normal conditions. The density of the corrosion current caused by hydrogen release ($i_{cor} = dQ/dt$) can be easily determined by differentiating the dependence of Q on t, where t is the residence time of the aluminum sample in solution.

The effectiveness of the inhibitor (citric acid monohydrate) was determined by its effect on the rate of hydrogen release on aluminum. All measurements were carried out in a 2 M KOH solution in 90% ethanol containing additives $Ga(NO_3)_3$, $In(NO_3)_3$ and various concentrations of citric acid monohydrate.

RESULTS AND THEIR DISCUSSION

Fig. 1 shows chronopotentiograms of galvanostatic discharge obtained on an aluminum electrode in a 2 M KOH solution in 90% ethanol containing 10^{-4} M Ga^{3+} and 10^{-3} M In^{3+}.

The displacement of the electrode potential towards positive values from the steady-state value of the open circuit potential at an anode current density of 4 mA/cm^2 does not exceed 50 mV. The stability of the potential value of the Al electrode during the discharge process should be noted. An increase in the water content in a mixed aqueous-ethanol solution from 4 to 10% leads to a fourfold increase in the achievable value of the discharge current density compared to what was previously obtained in a similar electrolyte containing 96% ethanol [15]. Fig. 2*a* shows the curves of the dependence of the amount of electricity spent on the release of hydrogen on aluminum, determined by the volume of the released gas, on the residence time of the Al sample in a solution containing various concentrations of the inhibitor. By differentiating the dependence of the amount of electricity Q on time t, the dependences of the corrosion current of aluminum i_{cor} on the time it was in the solution are obtained (Fig. 2b). As follows from Fig. 2*b*, the addition of citric acid monohydrate, depending on its concentration in the solution, leads to both a decrease and an increase in the density of the corrosive current. Citric acid monohydrate has the maximum inhibitory effect at a concentration of $5 \cdot 10^{-4}$ M in solution.

With higher and lower concentrations of citric acid monohydrate, the density of the corrosive current increases.

The corrosion current of aluminum, as can be seen from the data shown in Fig. 2, depends on the time it is in solution. The effectiveness of the inhibitor was evaluated using a steady-state value of the corrosion current. The corrosion inhibition efficiency η was calculated using the ratio $\eta = [(i_{oc} - i_{c}) / i_{oc}] \cdot 100$, where i_{oc} and i_c are the corrosion currents in the absence of the inhibitor and when it is in solution, respectively. At a concentration of citric acid monohydrate $5 \cdot 10^{-4}$ M, the inhibition efficiency calculated after a six-hour exposure of the aluminum sample in solution is 58%. The steady-state value of the corrosion current density is 1.7 mA/cm^2.

Fig. 3 shows chronopotentiograms of galvanostatic discharge of Al in a 2 M KOH solution in 90% ethanol containing additives of 10^{-4} M Ga^{3+}, 10^{-3} M In^{3+} and $5 \cdot 10^{-4}$ M citric acid monohydrate. A comparison

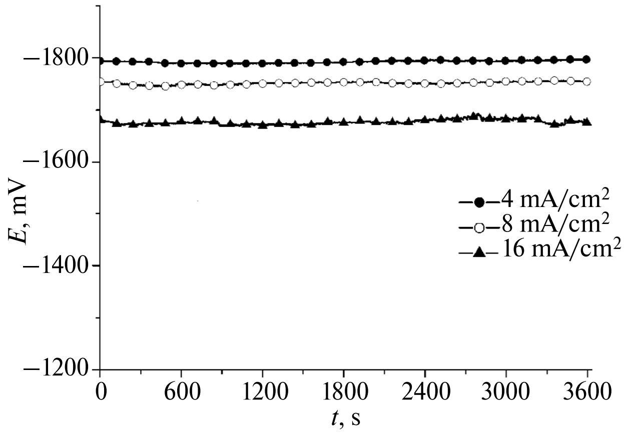

Fig. 3. Galvanostatic discharge curves on an Al electrode in a 2 M KOH solution in 90% ethanol containing 10^{-4} M Ga^{3+}, 10^{-3} M In^{3+} and $5 \cdot 10^{-4}$ M citric acid monohydrate at various current densities.

of the data shown in Fig. 1 and 3 shows that the introduction of citric acid monohydrate into the solution at a concentration of $5 \cdot 10^{-4}$ M does not impair the discharge characteristics of the aluminum electrode. Just as in the absence of an inhibitor, curves with a discharge current density of up to 16 mA/cm^2 demonstrate a stable discharge plateau.

CONCLUSION

1. An increase in the water content in a 2 M KOH solution in ethanol containing additives of gallium and indium compounds from 4 to 10% leads to a fourfold increase in the achievable value of the discharge current density.

2. Discharge galvanostatic curves in a 2 M KOH solution in 90% ethanol containing 10^{-4} M Ga^{3+} and 10^{-3} M In^{3-} at current densities up to 16 mA/cm^2 demonstrate a discharge plateau.

3. The introduction of citric acid monohydrate into the above solution at a concentration of $5 \cdot 10^{-4}$ M reduces the current density of aluminum corrosion to 1.7 mA/cm^2. The inhibition efficiency of citric acid monohydrate is 58%.

4. The introduction of citric acid monohydrate into the solution reduces the amount of aluminum corrosion current without reducing the rate of its anodic dissolution.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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