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Voltage Oscillations during Anodic Polarization of Zinc in Water Solutions of NaOH

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Abstract: The present brief research deals with the appearance of voltage oscillations during galvanostatic anodization of Zn in the NaOH electrolyte. Hence, this research aims to describe the observed voltage oscillations, giving the most probable explication for their occurrence. Following previous results, the anodization was performed at 30 or 50 mA•cm^{-2} in diluted 0.002 M NaOH aqueous solution for 100 or 240 s at room temperature. The respective voltage alterations were recorded and submitted for further analysis. The obtained curves and the probable reasons for the observed voltage oscillations were analyzed in detail. The main research result has become a concept for the correlation between the oscillations and the alteration of the growing layer resistivity. This suggests that the resistivity alters due to local heating effects inside the film. These effects probably cause irreversible structural changes in the bulk of the growing film, obviously related to the occurrence of oxygen vacations. In this sense, this study initiates an entirely new field of investigation regarding the phenomena related to the Zn anodization in alkaline media at high current densities.

Keywords: zinc anodization, voltage oscillations, conductivity, Joule heat.

氫氧化鈉水溶液中鋅陽極極化過程中的電壓振盪

摘要：目前的簡要研究涉及在氫氧化鈉電解質中對鋅進行恆電流陽極氧化過程中電壓振蕩的出現。因此，本研究旨在描述觀察到的電壓振盪，給出它們發生的最可能的解釋。根據先前的結果，陽極氧化在 30 或 50 毫安/厘米 2 下在稀釋的 0.002M 氫氧化鈉水溶液中在室溫下進行 100 或 240 秒。記錄並提交相應的電壓變化以供進一步分析。詳細分析了獲得的曲線和觀察到的電壓振蕩的可能原因。主要研究成果已成為振盪與生長層電阻率變化之間相關性的概念。這表明電阻率由於薄膜內部的局部加熱效應而改變。這些影響可能會導致大部分生長薄膜發生不可逆的結構變化，這顯然與氧氣休假的發生有關。從這個意義上說，這項研究開創了一個全新的研究領域，即高電流密度下鹼性介質中與鋅陽極氧化相關的現象。

关键词：鋅陽極氧化、電壓振盪、电导率、焦耳熱。

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1. Introduction

It is out of doubt that the anodic polarization of the zinc is essential for the researchers. This interest arises mainly because of the development of energy storage devices [1–12], but there are many other fields of application of zinc anodes. It is important to mention that the anodic films on zinc possess a great variety of chemical compositions and morphologies [13–22].

Several deviations from the “normal” behavior were observed for the anodic polarization of zinc. The dependence of the duration of the induction period on the concentration of the forming electrolyte for the anodizing of zinc in water solutions of oxalic acid is “opposite to the normal behavior.” Commonly, the duration of the induction period increases with an increase in the concentration, while for the zinc it decreases [23]. Another anomalous behavior is self-destroying of the film in water borate electrolytes [24]. Further, during other experiments, on the anodization of Zn in NaOH electrolyte [25], it was established that voltage oscillations appear at the higher current density values range. These oscillations were not commented in that work, and will be described in this brief study.

Hence, the aim of this research is to describe the observed voltage oscillations, giving the most probable explication for their occurrence.

2. Materials and Methods

The electrodes with area 2cm^2 were cut out of zinc foil (Alfa Aesar, 0.25 mm thick, purity 99.98%). The working surface was degreased with a 2% Nital and was further electropolished in an alcohol solution of phosphoric acid for 20 min at 20 V and temperature of 278 K according to [21] in a cell with a palladium cathode. The process of anodic polarization was carried out in a standard two-electrode cell with gold cathode without stirring to avoid hydrodynamic processes. Water solutions of NaOH with concentrations 8 mM•dm^{-3} and 2 mM•dm^{-3} were used as film formation electrolytes. The current densities were 30, 35, and 50 mA•cm^{-2}. The temperature was kept at 293 K.

3. Results and Discussion

A typical kinetic curve for zinc anodized in a water solution of sodium hydroxide is represented in Fig. 1. The difference between the “normal” kinetic and the present in Fig. 1 is the presence of point 1, in which the slope of the curve dU/dQ changes. It was called “bending point.” Before this point, the slope is greater than after it. The reason for the appearance of this change, and the dependence of the position of point 1 on the parameters of the anodic polarization were discussed in our previous work [25].

An interesting phenomenon was observed at high values of the current and the voltage (i.e. at small values of the formation electrolyte concentrations) in the vicinity of the bending point (Fig. 2).

The appearance of oscillations is observable around this point. These oscillations appear before this point (U_{bend}, Q_{bend}) and quench after it. It was also observed that at lower current density (30 mA•cm^{-2}), these oscillations are with lower amplitude and shorter, whereas at 50 mA•cm^{-2} are larger and more clearly expressed.

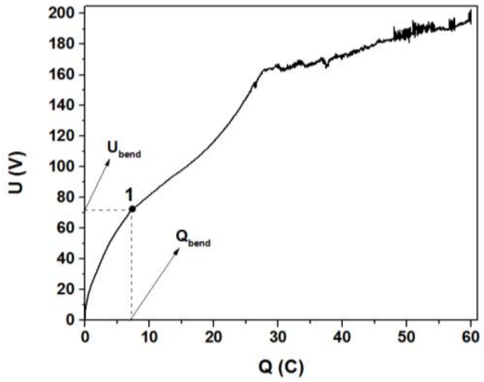

Fig. 1 A typical kinetic curve for zinc anodized in 8 mM•dm^{-3} water solution of NaOH with j = 35 mA•cm^{-2} at 293 K

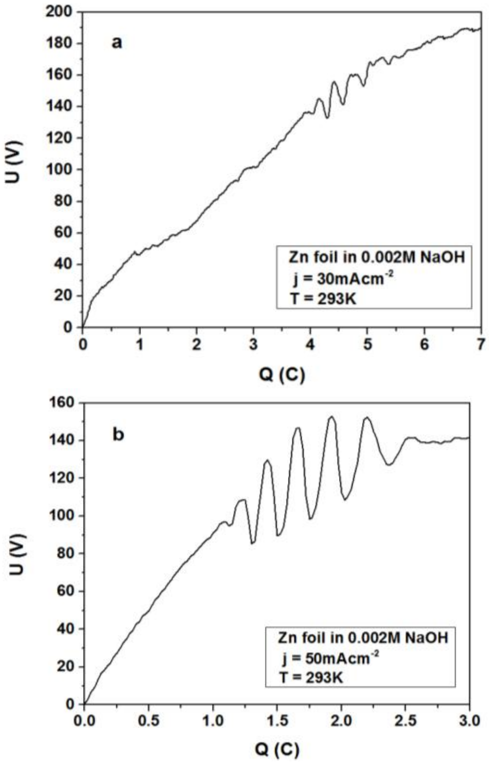

Fig. 2 A typical kinetic curve for zinc anodized in 8 mM•dm^{-3} water solution of NaOH with j = 35 mA•cm^{-2} at 293 K

The alteration in the anodic film conductivity at 305 K [25] can explain this difference that could be used as additional evidence for the hypothesis proposed in [25], that the change in the kinetic slope is a result of the change in the film conductivity, due to the temperature increase.

As was mentioned in [25], after reaching a defined thickness, the resistance of the growing film becomes rather high. As the main result, the Joule heat increases, resulting in an increment of the layer temperature. When the temperature inside the layer reaches 305 K, structural alterations become in its bulk. These alterations lead to decay of its specific resistance, which causes an increase in the slope of the kinetic curve. The generated heat does not allow temperature decrement, so that the growing layer maintains its specific resistance values (Fig. 1). At sufficiently high current and voltage values, the reaching of 305 K becomes at a relatively low layer thickness. When its specific resistance decays, the Joule heat decreases, as well. The film undergoes cooling and its temperature decays below 305 K. Hence, the newly grown layer possesses higher specific resistance. In turn, it promotes an increment of the Joule heat, resulting in reaching 305 K. Thus, the specific resistance decreases again and this cycle repeats, but at higher film thickness. Hence, after several cycles, the resulting resistance of the film becomes sufficiently high to maintain a temperature above 305 K. As result, the anodization process continues without voltage oscillations, and at lower specific resistance of the layer. Therefore the slope of the kinetic curve is smaller at the bending point.

Following this concept, the oscillations appear at lower potential values, when higher currents are applied. This fact is a consequence of the accelerated heat release at the application of higher currents. However, the heat dissipation remains at the same rate since the geometry of the system is the same, and the specific resistance alteration also appears at the same temperature. These effects are shown in Fig. 2.

4. Conclusion

This research is devoted to the appearance of voltage oscillations, during Zn anodization in alkaline medium at high current densities. Their occurrence was explained, suggesting an alteration of the specific resistance of the growing layer, due to localized temperature increment in its bulk. Further, it was established that the temperature elevation is a consequence of the Joule heat, resulting from the applied high current densities. Once a critical temperature of 305 K is reached, the film resistance decays, resulting in a temperature decrease. Hence, the main achievement of this study is exactly the description of this concept regarding the most probable mechanism of the observed voltage oscillations.

In this sense, this study initiates an entirely new field of investigations, regarding the phenomena, related to the Zn anodization in alkaline media at high current densities. The exact structural changes in the formatted layer will be an object of future research activities.

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