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Factors Affecting the Cathode Edge Nodulation in Copper Electrorefining Process

A. Ahmadi[a], S. Sheibani*[a], M. Mokmeli[a], S. M. J. Khorasani[b], N. S. Yaghoobi[c]

[a] School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran
[b] Process Control Unit, Khatoonabad Copper Refinery, Shahrebabak Copper Complex, Kerman, Iran
[c] Research and Development Center, Shahrebabak Copper Complex, Kerman, Iran

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ABSTRACT

In this study the factors that are affecting the copper nodular growth on the cathode edge were investigated from metallurgical and operation point of view. Statistical analysis was performed to evaluate the effect of operational conditions on the nodular copper growth by characterization of the nodule-containing cathodes. Besides, the effects of defects on polymer edge strips as well as changes in weight and thickness of anodes on the formation of nodules were investigated. Electrochemical galvanostatic experiments were employed to study the effect of electrolyte additives and the distance between the anode and cathode on cathode surface quality. A relatively large porosities of about 50 µm were observed in the microstructure of the cathode edge nodules. In addition, few nodule samples that were taken was observed to have a higher concentration of Fe, Cd and Pb, up to 25 ppm. Low probability (1%) in the repeatability of the nodule formation over the same position on the edge strip was approved the insignificant effect of possible edge strip defects on nodulation. The large weight variation of anodes can cause the anode thickness variation by 10 mm and consequently alter the distance between the anode and the cathode. This was shown to cause formation of nodules at the cathode edge. The peaks that were observed in the cathodic potential curves in galvanostatic tests, were believed to be the sign of nodulation and therefore was investigated further using the optical microscopic images.

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1. INTRODUCTION

Copper electrorefining is the final process of copper production. In this electrolytic process, impure copper (99.4-99.5%) as an anode, dissolves and electrodeposits on the surface of the cathode blank or starter sheet as pure copper cathode(>99.995%) [1,2]. Copper nodular growth as an unwanted phenomenon in copper electrorefining process, reduces the surface quality of the product and therefore its economic value [3,4]. Furthermore, the nodulation can causes an uneven distribution of the current density, a reduction in current efficiency, and hence, an increase in the operating cost of the refinery plant. Also, the short-circuiting caused by nodules adversely affect the current efficiency [5-7]. Copper nodular or dendritic growth is a complex phenomenon affected by different factors such as the physical parameters of a starter sheet or cathode blank and anode, electrolyte additives and temperature, circulation condition and chemical composition of the anodic slimes [8]. The electrolyte additives are organic reagents that are added to the electrolyte to prevent nodulation and control the physical properties of the cathode [9]. Nodulation was attributed to several factors including: (i) the inclusion of slime particles on the surface of cathode, (ii) the local shortage of leveling additives (Cl^-, thiourea and gelatin), and (iii) the deformation of the stainless-steel starter sheets. It is believed that once a nodulated surface develops, the localized current density, and hence the copper deposition rate, increases abruptly resulting in the further rapid growth of the nodules. So far, investigations of the nodulation have mainly been focused on the different effective parameters causing the nodulation and mechanisms of the nodules' formation [10,11]. Most of the nodules are grown on the cathode surface due to the electrolyte impurities including Pb, Se, Te, Ag, Bi, As

*Corresponding Author Institutional Email: ssheibani@ut.ac.ir (S. Sheibani)

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and Sn [12,13]. Other types of nodules may grow due to the operational factors such as variation in the distance of anode and the cathode [14]. One of the factors that can change the distance between the anode and the cathode that caused the nodulation is the surface quality of the anode and its weight [15]. The anode weight variation, affects the distance between the anode and cathode which is significantly affected the process [16].

The use of new materials and new characterization techniques is considered a special approach in materials engineering that has been considered in recent years to solve challenges [17,18]. The galvanostatic technique may be used as a helpful tool in detecting the formation of nodules on the cathode surface during the copper electrodeposition process using the value of the starting electrolytic potential and monitoring the cathodic polarization peak on a potential-time curve [19]. In previous studies [2,20] a correlation between the above-mentioned factors and nodular growth at the cathode surface was proven. To the best knowledge of the authors, no reports have studied the conditions at which the nodules may grow on the cathode edge and edge strips. Besides, the mechanism of nodular growth on the cathode edges is not understood well. The cathode edge nodule is a common unwanted phenomenon that more or less is observed in any copper refinery; that was investigated in this study. In this work, it is proven that the anode and cathode configuration is an important factor that affects the nodulation. This will be shown by changing the cathode and anode distance where a small variation in a call configuration largely affects the current density distribution and consequently a growth of irregular crystals. The edge nodulation can cause problems during the copper production process such as damaging the refinery equipment and the polymer edge strips and therefore reduces the production efficiency. This study aims to reveal the nodulation mechanism of the edge side and to evaluate factors that may fortify the edge nodulation using the electrochemical tools.

2. EXPERIMENTAL PROCEDURE

Samples were used in this study were all taken from a refinery plant of Khatoonabad copper complex. At first, nodule-containing cathodes were marked and sampled, then various analyses were performed on samples both with and without nodules, as shown in Figure 1. The microstructure was studied using an optical microscope (OLYMPUS-BH2UMA), a stereo microscope (NSZ-806) and a FESEM (Cam-Scan MV2300). Besides, the chemical composition and impurity analysis was performed using ICP-MS (ELAN DRC-2-Perkin Elmer SCIEX). In addition, the effect of various operational factors such as possible defects in the polymer edge strip or cathode blank and the changes in a weight and thickness of the anodes was statistically analyzed by taking different samples from the plant at different time intervals.

The nodules formation and growth was investigated using the galvanostatic technique. The electrolyte solution was made similar to Khatoonabad refinery plant. The electrolyte was made at a composition of 42 $g.L^{-1}$ $CuSO_4$, 160 $g.L^{-1}$ H_2SO_4, 40 $mg.L^{-1}$ HCl, 4 $mg.L^{-1}$ thiourea, and 11.5 mg L^{-1} gelatin. The electrolyte was preheated to 55 °C±0.5 °C. The current density used in the experiments was 500 $A.m^{-2}$. The deposition continued for 1 h without stirring. A commercially pure titanium plate (30×10×1 mm), a 316 stainless steel plate (30×12×3 mm) and anode copper plate (30×10×5 mm) from Khatoonabad were used as reference electrode, cathode and anode. The potential of the cathode surface relative to reference electrode was measured. Electrode surface preparation was performed using silicon carbide grinding papers following by cleaning, rinsing, and drying. The facial surface area of the cathode plate submerged in the electrolyte was 1.2 cm^2. All electrorefining experiments were performed at constant current densities using a rectifier (IPC SL20PRC). The galvanostatic tests were employed to investigate the effect of additives in the electrolyte as shown in Figures 2(a) and 2(b) and to investigate the effect of the anode and cathode arrangement on nodular formation. For this purpose, the effect of reducing the distance between the anode and cathode in various configurations were investigated (see Figure 2).

Figure 1. Samples of the cathode at different regions (a) without and (b) with nodules on the edge

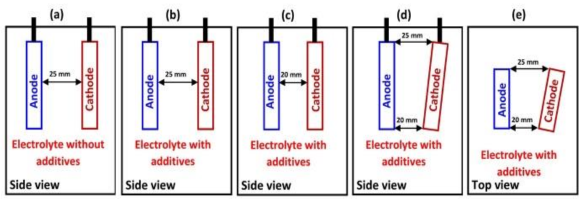

Figure 2. Position and distance of anode and cathode in the galvanostatic cell for experimental conditions of (a) T1, (b) T2, (c) T3, (d) T4 and (e) T5 experiments

3. RESULTS AND DISCUSSION

3. 1. Microstructure and Chemical Analysis of Nodules

Microscopic techniques have been used to examine and compare the microstructure of samples. Optical microscope and FESEM images of the cathode at different regions with and without nodules on the edge are shown in Figure 3. Comparison of the images for samples without and with nodules presented in Figures 3(a) and 3(b), respectively. That indicates the only noticeable difference to be the presence of relatively large porosities in a sample with nodules as shown in the optical and FESEM images in Figure 3(b). The size of the pores can easily reach up to 50 µm. Beside this large pore, both samples are contained very small porosities. This change in the microstructure can be caused by nodule formation or the cause of nodule formation, which will be further investigated.

Considering that the impurities can be a reason of nodule formation, the nodules at the cathode edge were sampled and analyzed. The chemical composition results are given in Table 1. The results showed similar values for most of the elements. However, for the few samples that were taken, differences in anlaysis of the three elements of Fe, Cd, and Pb was observed. The amount of Fe, Cd and Pb in the sample taken from the nodule was higher or equal to 22, 22 and 25 ppm, respectively; while for the sample without nodule were less than 1 ppm. This variation does not change the total concentration of the cathode and its deviation form cathode grade 1. The level of impurities that was proved to be the source of nodulation and was reported in the previous research [13], is much higher than the values found in the present study. For example, in presence of Pb up to 80 ppm, no nodule was observed in the cathode [13]. Therefore, it might be concluded that the presence of impurities at levels observed in the present study is not the main reason for nodule formation. It should be noted that this analysis is dependent on the number of analyzed samples.

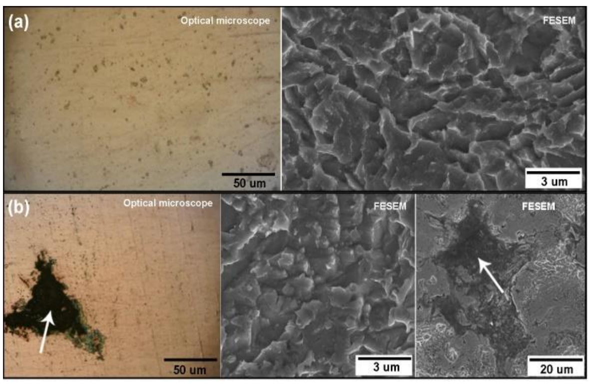

Figure 3. Optical microscope and FESEM images of the cathode at different regions (a) without and (b) with nodules on the edge

TABLE 1. ICP analysis results of the sample without and with nodules (wt.%)

Element	Sample without nodules	Sample with nodules
Cu	99.995	99.992
Ag	0.005	0.001
Ba	0.0001<	0.0001<
Co	0.0001<	0.0001<
Fe	0.0001<	0.0022
Mg	0.0001<	0.0001<
Pb	0.0001<	0.0025
P	0.0001<	0.0001
Si	0.0001<	0.0001<
Ti	0.0001<	0.0001<
Al	0.0001<	0.0001<
Bi	0.0001<	0.0001
Cr	0.0001<	0.0001<
Mn	0.0001<	0.0001<
Se	0.0001<	0.0001<
Cd	0.0001<	0.0022
Sn	0.0001<	0.0001<
V	0.0001<	0.0001<
As	0.0001<	0.0001<
Ca	0.0001<	0.0001<
K	0.0001<	0.0001<
Mo	0.0001<	0.0001<
Ni	0.0001<	0.0001<
Ga	0.0001<	0.0001<

One of the possible reasons for nodule formation at the cathode edge could be similar to the conventional nodules formation and due to the accumulation of impurities at the cathode edge. As a result, the reason for accumulation of impurities in this particular area must be determined. Beside impurity distribution, other factors such as operational factors should be studied.

3. 2. Effect of Operational Factors on Nodular Growth

In this section and according to the results obtained in Table 2, the effect of operational factors on the formation of nodules at the cathode edge was investigated. In this regard, the effect of factors that disrupt the current density distribution was investigated. These possible factors are as follows:

3. 2. 1. Physical Defects in the Polymer Edge Strip or Cathode Starter Sheets

To investigate the effect of possible defects in the polymer edge strip or cathode blank, the repeatability of nodule formation at marked locations of cathodes were evaluated.

This was performed on 60 cathodes of Khatoonabad copper refinery. For this purpose, the cathode blank were carefully marked at the nodulation place. After cathode stripping, the cathode blank were placed back in the electrolyte cell for another 10 days. The position of nodules at the edge of the cathodes was then determined and the repeatability of the nodule formation in each cathode and at the same position was monitored. The statistical results are summarized in Figure 4. The repeatability of nodule formation for a second time in a similar cathode blank was found to be 25%. The probability of recurrence of nodule formation in the similar position of a cathode blank was however below 4% (1 of 60 cases). Nodules in the remaining 21% were occurred elsewhere at the cathode edge. From these results, the conclusion can be drawn that nodulation on the polymer edge strip is not due to physical defects. Because if the defect in the edge strip caused nodule formation, the probability of repeating of nodule formation in the same previous position should be higher. For this reason, other possible factors were examined.

3. 2. 2. Changes in Weight and Thickness of Anodes Possible effect of changes in the anode weight and thickness on nodulation at the cathode edge, was studied by monitoring the weight distribution of 4000 anodes that is shown in Figure 5. The average anode weight was observed to be 346 kg. The minimum and maximum weights are 338 and 373 kg, respectively. In this analysis, the allowable number of changes in the weight of the anodes relative to the average weight was important. Previous research [18, 21] showed that the maximum tolerance up to ±10 kg have been accepted. The weight change can cause a thickness change of up to ±5 mm, depending on the dimensions of the anode. Considering the dimensions of the anodes used in Khatoonabad copper refinery, the relationship between thickness versus anode weight is shown in Figure 5. It can be seen the allowable range of weight change that results in a thickness change of less than 1 mm is about ±5 Kg. This range of weight change is shown on the

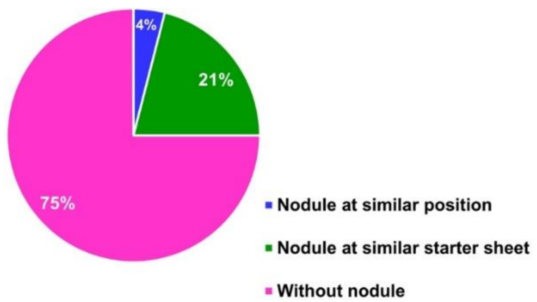

Figure 4. Results of the repeatability of nodule formation at a specific position on the cathode starter sheets

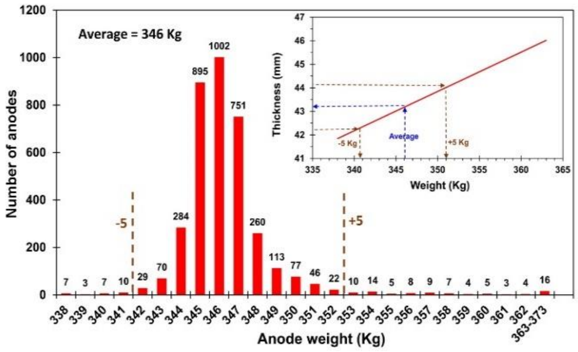

Figure 5. Distribution diagram of anodes weight. The inset shows the anode thickness versus anode weight

weight distribution diagram in Figure 5 with a dashed line.

As a result, approximately 15% of the anodes weigh is out of the acceptable range, and about 1% of the anodes weigh more than 20 kg of the average weight. This large weight difference can lead to a change in anode thickness up to 10 mm. It should be noted that this weight change often does not cause a uniform change in anode thickness. This change in the thickness of anodes is equal to a large change in the distance between the anode and the cathode in the cell, which can cause a change in the current density of the none uniform surfaces and nodule formation.

In Figure 6, the thickness of above-mentioned anodes were measured at different positions of the anode (top, bottom, left and right). In Figure 6 the thickness varies at different positions of an anode and as the weight of the anode increases, the thickness difference increases. For example, in the anode weighing 373 kg, the difference between the left and right side of the anode is 9 mm. This difference in thickness will change the distance between the anode and the cathode. For this reason, the current density will be different in these two positions. This phenomenon, especially at the beginning of the process, can cause anode passivation or nodular growth on the cathode in a position with smaller distance difference.

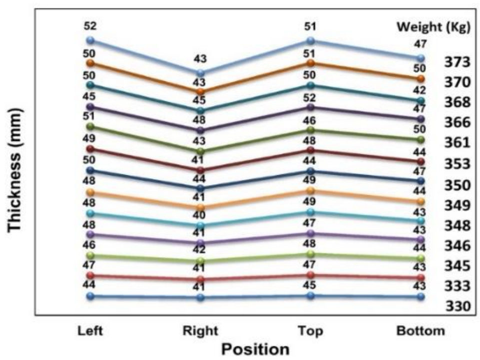

Figure 6. Changes of anode thickness in its various positions (left, right, top and bottom)

Previous research [18, 21] showed that a 5 mm local reduction in the distance between the anode and cathode can increase the current density by up to 20% locally. The distance at the anode edges is therefore a reason for nodule formation at the left side cathode edges.

3. 3. Galvanostatic Tests Results According to the previous section observation, galvanostatic tests were performed to investigate the effect of nodule formation factors in a laboratory scale. According to the anode and cathode arrangement presented in Figure 2, the cathodic potential curves versus time were generated and showed in Figure 7. No peaks were observed for the arrangement b, T2 experiment, while two or three peaks are seen for other arrangements. In each peak, the cathodic potential decreases by approximately 40 to 60 mV. The presence of the peak in the cathodic potential curve is an indication of current unbalance during the deposition process. Based on the previous research [19, 20, 22], this current unbalance can be attributed to the nodular growth at cathode surface or edges. This means that for a test where additives is not used (T1 experiment), and for tests the cathode and anode have not normal arrangement, reducing the distance between the anode and the cathode from 25 to 20 mm (T3 experiment), either from the bottom (T4 experiment) or the lateral side (T5 experiment) have caused peak generation and possibly conditions for nodule formation. It should be noted that T2 experiment was developed similar to the industrial cell condition. A comparison of the T1 and T2 curves shows that the presence of additives in electrolyte increases the cathodic potential during the experiment [5, 6, 23].

The surface quality of cathodes formed at different conditions of galvanostatic tests was studied using an optical microscopic image shown in Figure 8. Images with higher magnifications from area 1 and 2 (shown in white rectangles) are shown at the right-hand side. Relatively smooth and homogeneous deposit without nodular growth was produced under T2 arrangement; as shown in Figure 8(b). However, in the absence of electrolyte additives (T1 experiment), the quality of deposit was not uniform and nodules were formed at the surface and edges of cathode; as shown in Figure 8(a). The additive role in improving cathode quality was verified in many previous studies [23, 24]. Additives promote the plating of smooth, dense copper deposits with minimal impurities. The effects of uniformly reducing the distance between the anode and cathode from 25 to 20 mm (T3 experiment), either from the bottom side (T4 experiment) or the lateral side (T5 experiment) is shown in Figures 8(c), 8(d) and 8(e), respectively. Reducing the distance has led to the formation of different types of nodules on the surface or on the edge. The results from the experiments indicate the importance of anode and cathode configuration in the cell. To produce a smooth cathode without nodules it is necessary to have a uniform distribution of current density over the entire cathode surface.

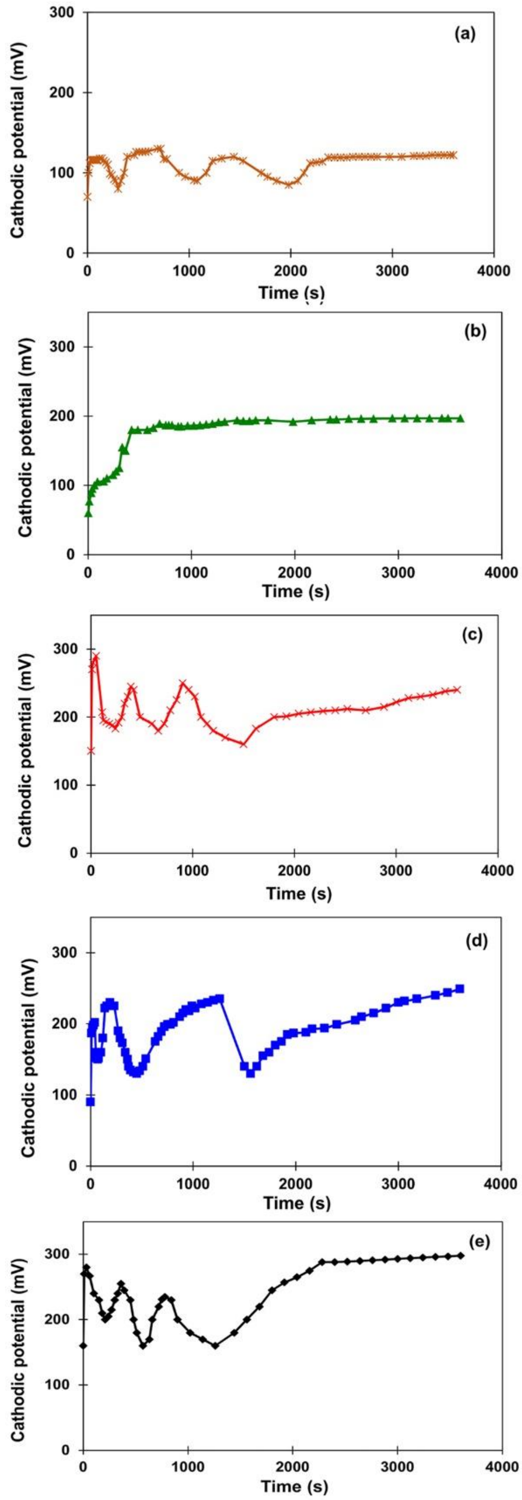

Figure 7. Cathodic potential curves versus time for (a) T1, (b) T2, (c) T3, (d) T4 and (e) T5 experiments

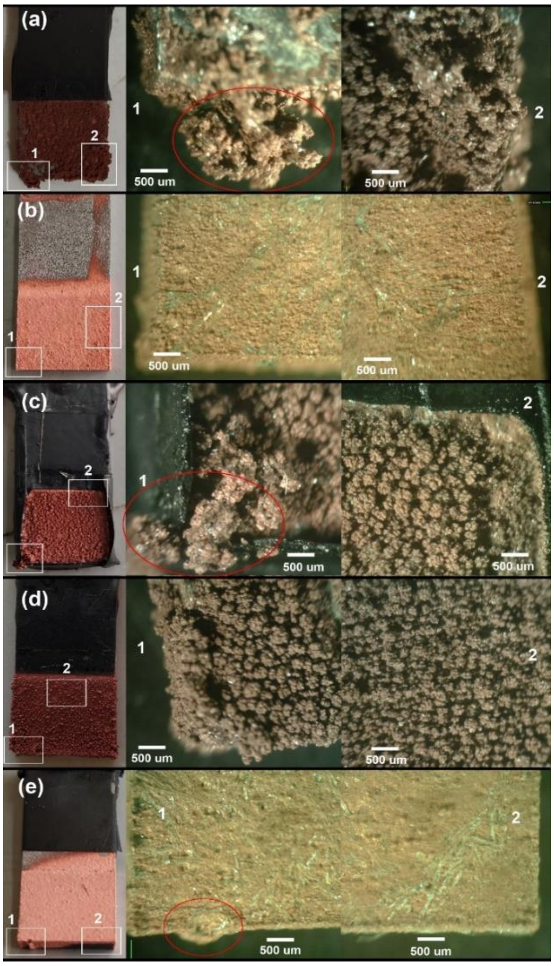

Figure 8. Optical images from the cathode surface for (a) T1, (b) T2, (c) T3, (d) T4 and (d) T5 experiments

To reach this objective, it is important to provide a constant distance between the anode and the cathode. It is also necessary to control the distance during the electrolysis operation. Ohmic electrolyte resistance, is dependent to the electrode distance and geometry. Different anode-cathode configuration with the consequence of variation in a current density resulted in irregular crystal growth and dendrite formation. Nodules on the edge were marked with a red oval in Figures 8(a), 8(c) and 8(e). The presence of peaks in the cathodic potential diagrams is consistent with nodule formation on cathode. In conclusion, changing the distance between the anode and the cathode not only caused the formation of nodules at the cathode edge but also at the cathode surface.

4. CONCLUSION

In this research, edge nodulation of the copper cathode was studied at Khatoonabad copper refinery. Investigation was conducted by looking into the galvanostatic experiments to investigate the nodular growth during electrorefining process. The main results are as follows:

1. The optical and FESEM images showed that the main microstructural difference between the nodule and nodule-free regions is the presence of relatively large porosities with diameter of up to 50 µm.
2. The difference between the chemical composition of the noduled area and nodule-free area for the samples taken was observed to be higher for Fe, Cd and Pb in the noduled samples. This change was not caused devation of total concentration from cathode grade 1.
3. The repeatability of nodule formation in a similar position of a starter sheet was found 4%. Hence, nodule formation was not due to the physical defects in the polymer edge strip or stainless-steel cathode blank.
4. A large weight difference can lead to a change in a thickness of anode and therfore be a source of nodule.
5. The galvanostatic test showed that reducing the distance between the anode and the cathode can cause the formation of nodules at the cathode edge and cathode surface. This is parallel with the presence of peaks observed in the cathodic potential experiments.
6. Changes in anode weight and consequently the thickness of the anodes can shorten the local distance between the anode and the cathodeand a reason for nodule formation at the cathode edges.

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Persian Abstract

چکیده

در این مطالعه، عوامل موثر بر رشد ندولار مس روی لبه کاتد از دیدگاه متالورژی و عملیاتی بررسی شده است. به منظور ارزیابی اثر شرایط عملیاتی روی رشد ندولار مس، تحلیل آماری روی کاتدهای دارای ندول انجام شده است. همچنین، تاثیر عیوب روی نوار لبه پلیمری و تغییرات وزن و ضخامت آندها بر تشکیل ندول مطالعه شده است. به منظور مطالعه تاثیر افزودنی‌های الکترولیت و فاصله بین آند و کاتد بر روی کیفیت سطح کاتد، از آزمایش‌های گالوانواستاتیکی الکتروشیمیایی استفاده شده است. تخلخل‌های نسبتا درشت با اندازه حدود ۵۰ میکرون در میکروساختار مربوط به ندول‌های لبه کاتد مشاهده شد. علاوه بر این، نتایج نشان داد که نمونه‌های ندول گرفته شده حاوی مقادیر بیشتری از آهن، کادمیم و سرب تا ۲۵ ppm بود. احتمال کم (۱٪) در تکرار تشکیل ندول در موقعیت یکسان روی نوار لبه بیانگر آن بود که تاثیر عیوب نوار لبه بر تشکیل ندول ناچیز است. تغییرات وزنی زیاد در آندها منجر به تغییرات ضخامت تا ۱۰ mm شده و لذا تغییر فاصله بین آند و کاتد را به دنبال دارد. این موضوع تشکیل ندول‌ها در لبه کاتد را باعث می‌شود. پیک‌های مشاهده شده در منحنی‌های پتانسیل کاتدی در آزمون‌های گالوانواستاتیکی می‌تواند به دلیل تشکیل ندول باشد که با استفاده از تصاویر میکروسکپی نوری مورد ارزیابی بیشتر قرار گرفته است.