

Optimization of Environment-friendly Electrolytic Polishing of Nitinol Stent

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Increasing number of patients with cardiovascular diseases continuously expands the market demand for stents. An environmentally friendly and nontoxic electrolytic polishing solution for stents is proposed to improve the surface quality of the laser cutting stent, eliminate slag, and recast layer. The best ratio of electrolyte is determined through experiments. On this basis, the importance of electrolytic polishing parameters was studied and optimized, and the best electrolytic polishing parameters were obtained. At the same time, the material removal rate, microscopic morphology, and surface roughness of the scaffold were investigated, and the removal rate of the bridge width and wall thickness of the scaffold was obtained. After polishing, the surface of the stent was smooth, without slag and recast layer, and the surface roughness reached approximately 10 nanometers.

Keywords: Nitinol stent; Environmentally friendly electrolyte; Electrolytic polishing parameters; Surface morphology

1. INTRODUCTION

Cardiovascular disease is the world's number one health killer, accounting for nearly 29% of the total deaths worldwide [1]. In 2018, the number of patients with cardiovascular disease worldwide reached 440 million; approximately 290 million patients have cardiovascular disease in China, and the number continues to increase [2]. In addition, cardiovascular disease is the most expensive disease in the United States. The direct medical cost in 2016 was 555 billion U.S. dollars. By 2035, this number is expected to double to 1.1 trillion U.S. dollars [3]. Heart stents account for 80% of the cost of cardiovascular disease treatment. The continuous increase in the number of patients with cardiovascular

diseases continuously expands the market demand for the stent industry. However, the material properties and processing quality of cardiovascular stents are highly in demand [4,5].

Heart stent, also known as coronary stent, is a commonly used medical device in interventional heart surgery [6]. It has the effect of dredging arteries. Common materials are stainless steel, nitinol, and cobalt–chromium alloy [7,8]. Nitinol is favored because of its shape memory, flexibility, and mechanical properties similar to human tissues [9,10]. The stent is small in size and has a complex hollow structure. The traditional mechanical cutting manufacturing process is very difficult, and it produces strong work hardening and excessive tool wear [11–14]. Laser cutting can avoid these difficulties. Its basic mechanism is thermal melting (sublimation) of the processed metal to achieve material removal [15]. Its disadvantage is that when the melted material is resolidified, part of the material solidifies on the support to form a recast layer and slag because it cannot be taken away by the cooling water or airflow [16,17]. In addition, a certain thickness of the heat-affected zone is generated in the cutting area due to the thermal cutting effect [18]. Therefore, postprocessing of the stent after laser cutting is required. Electrolytic polishing slightly damages the stent and has high polishing efficiency. It can remove the slag and recast layer produced by laser processing and improve the surface morphology of the stent [19,20].

Eun et al. studied the effect of two electrolytic polishing solutions on the surface roughness of nitinol plates. One type of electrolytic polishing liquid is composed of sodium nitrite and sodium tartrate, and the other type of polishing liquid is composed of sulfuric acid, phosphoric acid, and water. Experiments show that the first electrolytic polishing solution leads to fast material removal process; but, it produces many ultramicropores on the surface of the alloy and obtains poor metal morphology. The second polishing solution can obtain relatively good surface roughness (0.31 μm), which is more suitable for precision machining [21]. N.I.D.A. Lopes et al. used 3.5 mol/L methanol and sulfuric acid electrolyte to electrolytically polish nitinol wires. The results show that the surface roughness of the polished nitinol wire is effectively reduced (to approximately 0.1 μm), the nickel-rich layer on the surface is removed, and the corrosion resistance of the sample is improved [22]. H. Deokhyun et al. used methanol and sulfuric acid electrolyte to polish the drawn nitinol wire. Studies have shown that the surface quality of the sample depends on the electrolytic polishing parameters and is most sensitive to current density. The best combination of surface morphology and roughness (17.4 nm) can be obtained using the best experimental parameters [23]. H. Ji et al. polished the nitinol tube by using an electrolytic polishing solution of perchloric acid, methanol, and additives and analyzed the degree of influence of different polishing factors on the quality of the sample. The average surface roughness of the tube is 48.6 nm when using the best polishing process parameters. The surface morphology and roughness of the sample have been improved remarkably [24].

In the current literature, the research objects are mainly nitinol plates and nitinol wires. Studies on the surface modification and optimization of the nitinol stent are few due to the small size of the stent and the complicated hollow structure. The stent and the plate are remarkably different, and the process research on the plate is evidently unsuitable for the stent. In addition, in most studies, the main components of the electrolytic polishing solution used by the researchers are acid electrolytes (perchloric acid, sulfuric acid, and nitric acid). The follow-up treatment of this electrolyte is complicated, and it easily damages the staff and equipment. Therefore, the objective of this study consists of four parts. (1) Nitinol stents are used as research objects. (2) The environmentally friendly electrolyte is used as the

basic electrolytic polishing fluid, and the best ratio of additives is explored. (3) The influence of other electrolytic polishing parameters on the stent is analyzed and optimized. (4) A comparative study on the removal rate, microscopic morphology, and roughness of the scaffold before and after polishing is conducted.

2. EXPERIMENTAL

A nitinol tube (Ti47.5at.%, Luhao Metal Group Co., Ltd.) with an outer diameter of 2.62 mm at room temperature is used. The wall thickness of the tube is 0.21 mm. TX Plu model automatic laser cutting machine (Hongshi Laser Technology Co., Ltd.) is used to process the nitinol tube into a stent. The laser processing parameters are as follows: the laser power is 125 W, the pulse width is 13 us, the cutting speed is 5 mm/s, and the pulse frequency is 10000 Hz. The length of the stent after processing is 10 mm. The inner wall of the stent is polished with a wire file with a diameter of 1.5 mm to remove the slag that is not tightly connected to the substrate. The outer surface of the support is mechanically polished with #500-2000 SiC paper. Then, acetone is used to ultrasonically clean for 180 s, blow dried, and stored in an argon sealed tank. Figure 1 shows the microscopic morphology of the scaffold under the field emission environment scanning electron microscope (FESEM). Figure 1a shows a low magnification stent image.

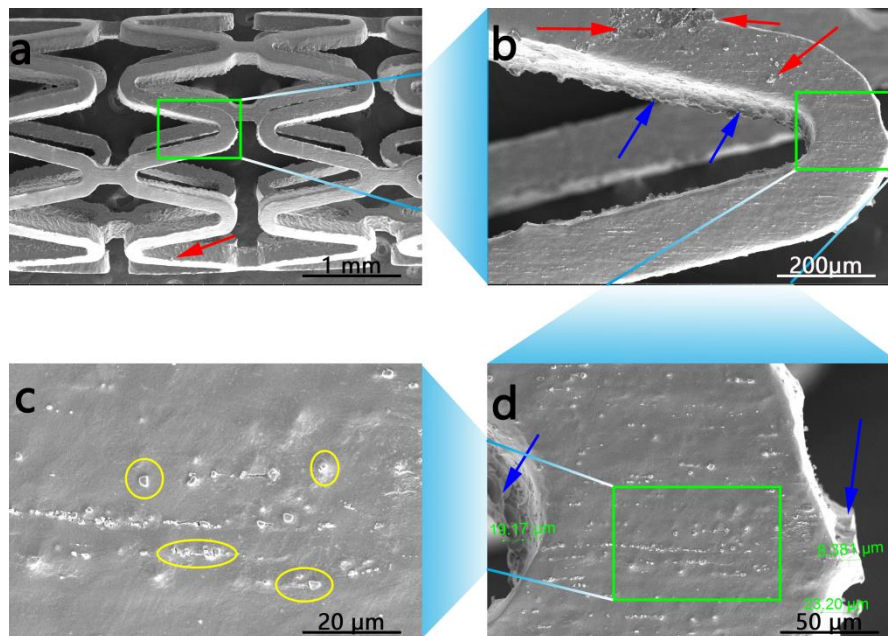


Figure 1. Surface micromorphology of the laser cut stent: (a) 100× (b) 500× (c) 5000× (d) 2000×

Figures 1b–d are partial enlarged views of Figure a. The figures clearly show that after laser cutting, large amounts of slag (red arrow) and recast layer (blue arrow) are attached to the surface of the

stent. Fig. 1d shows that the maximum thickness of the recast layer is $23.20\ \mu\text{m}$. In other locations, the average thickness of the recast layer is approximately $16.68\ \mu\text{m}$, which does not exceed $25\ \mu\text{m}$. In addition, the surface of the stent has many bumps and other uneven areas (yellow ellipses). The FE-SEM image shows that the surface quality of the stent is very poor, with many defects. The roughness of the stent is measured with an optical profiler (MicroXAM-100), and Ra is selected as the evaluation parameter. Three cardiovascular stents are selected for measurement, each of which is measured six times, and the measurement is performed at a different position of the cardiovascular stent each time. Then, the measured roughness is averaged as the final result. The results show that the surface roughness of the cardiovascular stent is $1.42\ \mu\text{m}$.

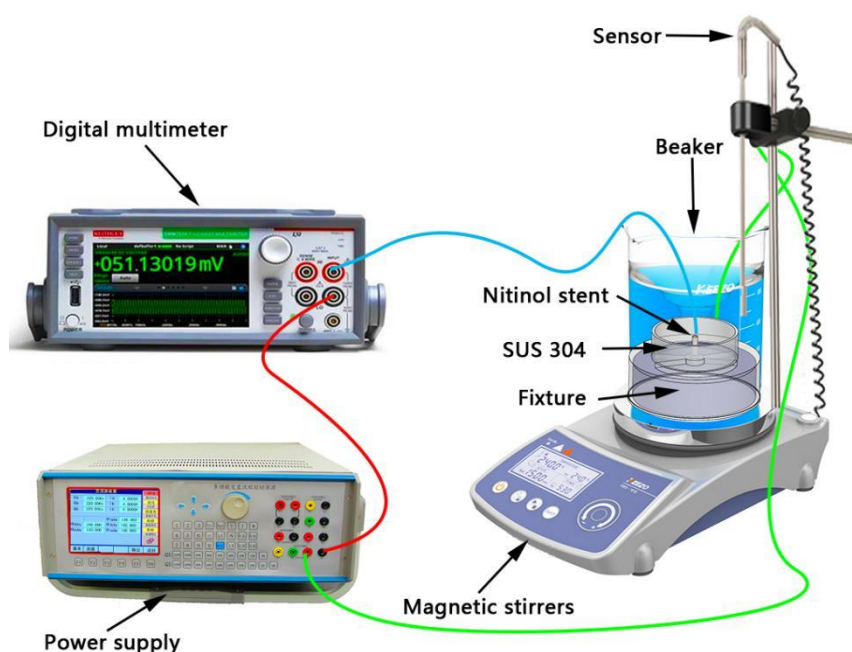


Figure 2. Schematic of the electrolytic polishing experiment platform of the nitinol stent

Table 1. Electrolytic polishing parameter range of nitinol stent

Voltage (V)	Electrolyte		Time (min)	Temperature (°C)	Distance between electrodes (mm)
	Basic electrolyte	Additive (ml/L)			
6-36	1mol/L sodium chloride-ethylene glycol (70% volume fraction) and ethanol (30% volume fraction)	Saturated potassium chloride solution 5-35	1-21	5-35	10-30

Figure 2 shows the schematic of the electrolytic polishing experiment used in the experiment. The stent is used as the anode, and the wire is connected to the anode of the DC power supply through the digital multimeter. Grade 304 stainless steel plate (STS304) is used as the cathode, which is connected to the negative electrode of the DC power supply. Electrolyte, stents, and fixtures are placed in the beaker. Industrial ethylene glycol (99% purity 1 wt% H₂O) was mixed with NaCl to form a 1 mol/L sodium chloride-ethylene glycol electrolyte solution. Anhydrous ethanol with a purity greater than 99.9% was added. Thus, the concentration of anhydrous ethanol accounted for 30% of the volume fraction of the electrolyte solution. The magnetic stirrer is used to speed up the circulation and temperature control of the electrolyte. According to the author's previous research and preliminary experiments, the range of electrolytic polishing parameters is as shown in Table 1 [25]. The quality of the polished stent can be greatly improved under such polishing parameters.

An optical profiler (MicroXAM-100, Shanghai Micro-Nano Metrology Technology Co., Ltd., China) was used to obtain the surface roughness (Ra) and the width and wall thickness of the support bridge. When measuring the size, measurement was performed at six different positions on each bracket; measuring was performed thrice per position. The average of the measured values was obtained. The field emission environmental scanning electron microscope (FE-SEM, FEI Quanta 250, America) was used to study the surface morphology. After several tests, the average wall thickness reduction was divided by the polishing time to obtain the material removal rate.

3. RESULTS AND DISCUSSION

3.1 Determining the electrolytic polishing solution

The composition of the electrolytic polishing solution is a key factor that affects the polishing quality. The ideal electrolytic polishing solution results in a smooth and flat surface of the workpiece and improves the processing efficiency. The polishing liquid mainly acts as a conductive medium to transfer current and cause electrolysis reaction under the action of the electric field to remove the material on the surface of the workpiece. The ideal electrolytic polishing solution should have high etching speed, solubility, and conductivity; the product that reacted with the anode is soluble salt, which is easy to handle. The introduction of the concept of environmental protection in recent years should conform to green manufacturing and focus on environmental protection.

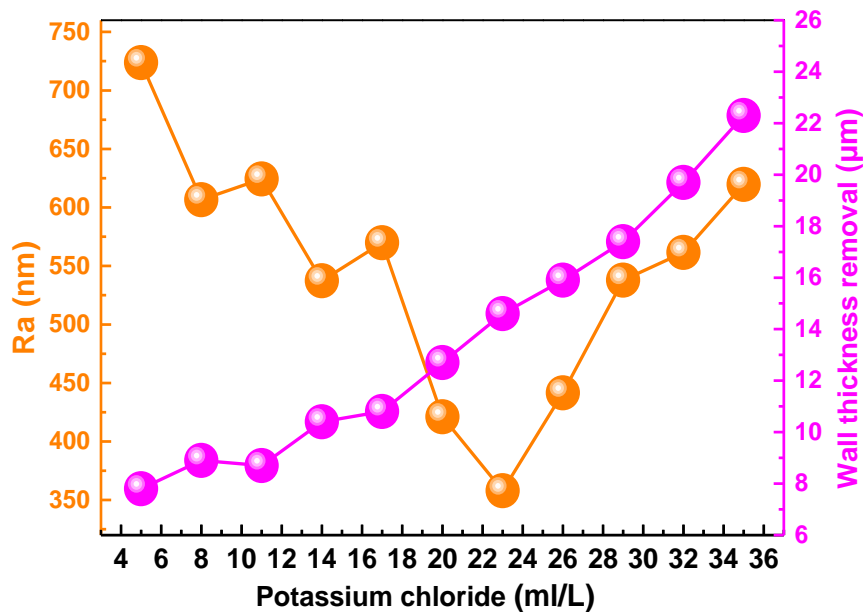


Figure 3. Influence of potassium chloride content (5–35 ml/L) on the wall thickness removal and surface roughness of the stent

Therefore, the author proposed an environmentally friendly electrolyte solution, namely, absolute ethanol (30% wt.%) and sodium chloride-glycol electrolyte solution. Polishing for 20 minutes using the polishing liquid can obtain better surface quality. The removal rate of the materials is increased to improve the efficiency of electrolytic polishing when using this polishing solution. According to the author's previous research and preliminary experiments, the preliminary selection of electrolytic polishing parameters are as follows: absolute ethanol (30% wt.%) and sodium chloride (1 mol/L)-ethylene glycol basic electrolytic polishing solution, saturated potassium chloride 5–35 ml/L (additive), polishing time 9 min, pole spacing 25 mm, temperature 20 °C, and voltage 20 V. The microscopic morphology and surface roughness of the stent are used as indicators to judge the quality of the parameters. The experiment uses saturated potassium chloride as a variable, with a tolerance of 3 ml/L, which is increased in accordance with the arithmetic sequence. Each group of variables is subjected to parallel experiments with three workpieces to ensure accuracy. After the experiment, the stent is cleaned and dried, and the wall thickness removal and surface roughness of the stent are measured. The result is shown in Figure 3.

Figure 3 shows that as the potassium chloride content increases, the surface roughness of the stent presents a double W-shaped law. When the potassium chloride content is 5 ml/L, the average surface roughness of the stent is 723.7 nm, which is 696.3 nm lower than the roughness of the stent prior to polishing. As the potassium chloride content continues to increase, the surface roughness of the stent generally tends to gradually decrease. The stent obtains the minimum roughness (357.9 nm) until the potassium chloride content reaches 23 ml/L. However, as the potassium chloride content continued to increase, the roughness of the stent begins to gradually increase. When the content is 35 ml/L, the roughness is 539.7 nm, indicating an increase of 50.80% compared with the minimum roughness. In

addition, the measured wall thickness removal curve shows that as the potassium chloride content increases, the wall thickness of the stent gradually decreases. This finding is due to the increase in potassium chloride, thereby increasing conductive ions in the electrolyte. Thus, the reaction between the anode and the cathode is accelerated, and the removal rate of the material is increased. When the potassium chloride content is 5 ml/L, the wall thickness is reduced by 7.8 μm . When the minimum roughness is obtained, the wall thickness of the stent is reduced by 14.6 μm . At this time, the removal of the wall thickness is less than the average thickness of the recast layer (16.68 μm). This finding indicates that the recast layer on the surface has not been completely removed under this parameter, and the parameters of electrolytic polishing need to be further optimized.

3.2 Optimization of electrolytic polishing parameters

The analysis results show that 23 ml/L of saturated potassium chloride solution is added to anhydrous ethanol (30% wt.%) and sodium chloride (1 mol/L) ethylene glycol basic electrolytic polishing solution. We can obtain a nitinol stent with a surface roughness of 357.9 nm. The surface quality of the stent has been improved to a certain extent under this parameter. However, the surface roughness is still extremely large, and the recast layer has not been completely eliminated. Therefore, further optimizing the parameters of electrolytic polishing time, temperature, voltage, and electrode spacing is necessary. The orthogonal experiment can obtain scientific experimental conclusions with less experiment time [26]. Therefore, we design the corresponding orthogonal experiment table (four factors and three levels), as shown in Table 2.

Table 2. Factors and levels used in the orthogonal experimental design.

Level	Polishing time (min)	Distance between electrodes (mm)	Voltage (V)	Electrolyte temperature ($^{\circ}\text{C}$)
1	3	13	8	10
2	9	19	20	20
3	15	25	32	30

After the test, the surface roughness of the stent under different parameters is measured, as shown in Table 3. Table 4 provides the evaluation indices of this orthogonal experiment, in which K_i ($i = 1, 2, 3$) and R are important parameters. K_i is defined as the average value of the surface roughness R_a at the j ($j = 1, 2, 3$) level of the corresponding factor f (f = polishing time, distance between electrodes, voltage for polishing, electrolyte temperature), and R is defined as the difference between the K_{\max} and K_{\min} values in the corresponding factor column. For example, when the factor f = Polishing time is 3 min, $K_1 = (639.2 + 449.6 + 507.3)/3$, $R = \max\{532.03, 386.80, 465.53\} - \min\{532.03, 386.80, 465.53\} = 145.23$.

By comparing K values, the optimal factor level can be obtained. The R value is an important analysis indicator, which indicates the fluctuation range of the K value. Therefore, the larger the R value, the more important this factor is [27,28]. From Table 4 combined with Table 2, we can see that the parameters for the minimum roughness under various factors are as follows: polishing time 9 min, distance between electrodes 25 mm, voltage 20 V, and electrolyte temperature 20 °C. In addition, Table 4 shows that the degree of influence on the surface quality of the stent in descending order is polishing time (R 145.23), voltage (R 99.90), distance between electrodes (R 64.50), and electrolyte temperature (R 40.17). Among them, the electrolytic polishing time has the greatest impact on the surface quality of the stent, and the electrolyte temperature has a relatively small impact on its quality.

Table 3. Test plan and roughness results

Test	Polishing time (min)	Distance between electrodes (mm)	Voltage (V)	Electrolyte temperature (°C)	Surface roughness Ra (nm)
1	3	13	8	10	639.2
2	3	19	20	20	449.6
3	3	25	32	30	507.3
4	9	13	20	30	378.5
5	9	19	32	10	392.6
6	9	25	8	20	389.3
7	15	13	32	20	480.7
8	15	19	8	30	507.6
9	15	25	20	10	408.3

Table 4. Results of roughness analysis

Parameters	Polishing time	Distance between electrodes	Voltage for polishing	Electrolyte temperature
K ₁	532.03	499.47	512.03	480.03
K ₂	386.80	449.93	412.13	439.87
K ₃	465.53	434.97	460.20	464.47
R	145.23	64.50	99.90	40.17

The surface quality of the nitinol stent should be improved to further optimize the electrolytic polishing parameters. On the basis of the conclusions of these orthogonal experiments, we conduct single-factor experiments on the electrolytic polishing factors in accordance with the degree of influence. The experiment was repeated three times for each experiment parameter. After the experiment, the stents for the three experiments were removed, washed, and dried, and then the instrument was used to measure the roughness. Each measurement is performed at six different positions of the stent, and each position is measured repeatedly three times. The average value of the three measurements is considered the roughness value.

3.2.1 Optimization of polishing time

According to Table 4, the range of polishing time is the largest among the four polishing parameters. Therefore, an experimental study on its influence law is initially conducted. The experiment uses electrolytic polishing time as a single variable, and the polishing time ranges from 1 min to 21 min. The remaining influencing factors are based on the orthogonal optimization parameters obtained in Tables 3 and 4 and are tentatively set as the distance between the electrodes of 25 mm, the voltage of 20 V, and the temperature of the electrolyte at 20 °C. After the experiment, a box plot of the influence of different polishing time on the surface roughness of the stent is drawn, as shown in Figure 4.

Figure 4 shows that the surface roughness of the stent initially decreases and then increases as the polishing time increases. When the polishing time is 1 min, the minimum surface roughness of the stent is 815.92 nm, the maximum roughness is 1018.44, and the average roughness is 926.02. The minimum, maximum, and average roughness values gradually decrease as the polishing time increases. When the polishing time is 11 min, the minimum, maximum, and average surface roughness of the stent are 239.84, 358.53, and 311.38 nm, respectively. Compared with the polishing time of 1 min, the roughness is reduced by 70.60%, 64.80%, and 66.37%. At this time, the surface roughness of the stent achieves a minimum. The roughness gradually begins to increase as the polishing time is extended again. When the polishing time is 21 min, the minimum, maximum, and average roughness are 640.66, 999.424, and 809.58 nm, respectively. Compared with the best polishing time, it is increased by 1.67, 1.79, and 1.60 times. Therefore, the best electrolytic polishing time is 11 min.

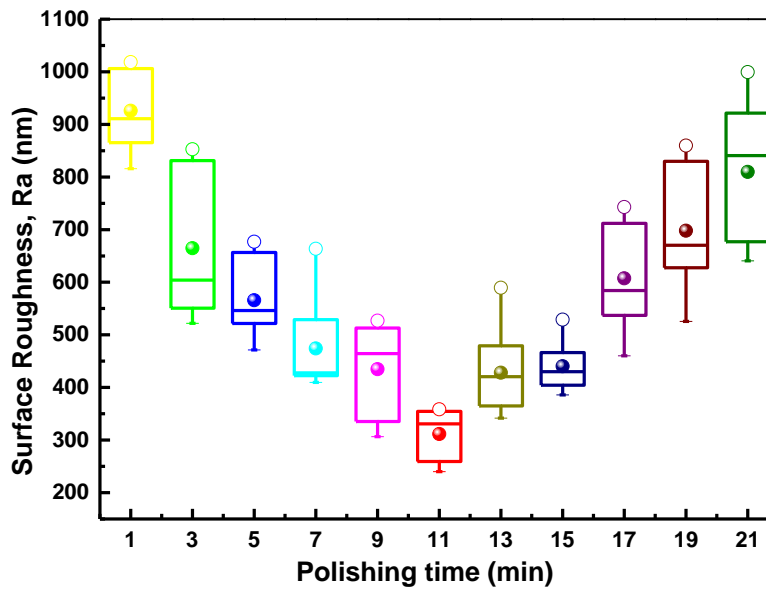


Figure 4. Box plot of stent surface roughness for different polishing time (1-21 min)

3.2.2 Optimization of voltage for polishing

Similarly, Table 4 shows that the voltage for polishing is the second most important factor affecting the quality of the stent in the polishing parameters. Therefore, the optimization of its parameters is ranked second. The experiment uses the voltage for polishing as a single variable, with a value range of 6–36 V [29]. According to the conclusion in Section 3.2.1, the best polishing time is 11 min. The remaining parameters are based on the orthogonal optimization parameters obtained in Tables 3 and 4 and are tentatively set as the distance between electrodes of 25 mm and the electrolyte temperature of 20 °C. After the experiment, the box plot of the influence of different voltages for polishing on the surface roughness is shown in Figure 5.

Figure 5 shows that with the gradual increase in the voltage for polishing, the minimum, maximum, and average roughness of the stent surface show a change rule that initially decreases and then increases. When the voltage for polishing is 6 V, the minimum, maximum, and average surface roughness of the stent are 701.69, 926.98, and 808.83 nm, respectively. Although the surface roughness is greatly reduced compared with the surface roughness prior to polishing, the roughness is still relatively large. With the gradual increase in the voltage for polishing, the minimum, maximum, and average roughness values gradually decrease. When the voltage for polishing is increased to 24 V, the minimum, maximum, and average surface roughness of the stent are 112.95, 213.97, and 155.48 nm, respectively. Compared with the voltage for polishing of 6 V, the roughness is reduced by 83.90%, 76.92%, and 80.78%. At this time, the surface roughness of stent is the minimum. As the voltage for polishing increases again, the roughness of the stent gradually increases. When the voltage for polishing increases to 36 V, the minimum, maximum, and average roughness are 519.7, 748.01, and 670.53 nm, respectively. Compared with the best polishing time, the corresponding increase is 2.60, 2.50, and 3.31 time. Therefore, the best electrolytic voltage for polishing is 24 V.

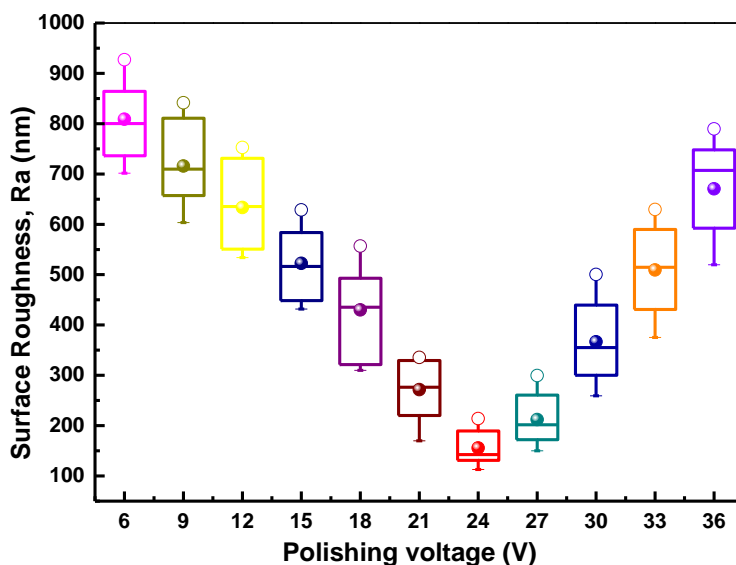


Figure 5. Box plot of stent surface roughness for different voltage for polishing (6–36 V)

3.2.3 Optimization of the distance between electrodes

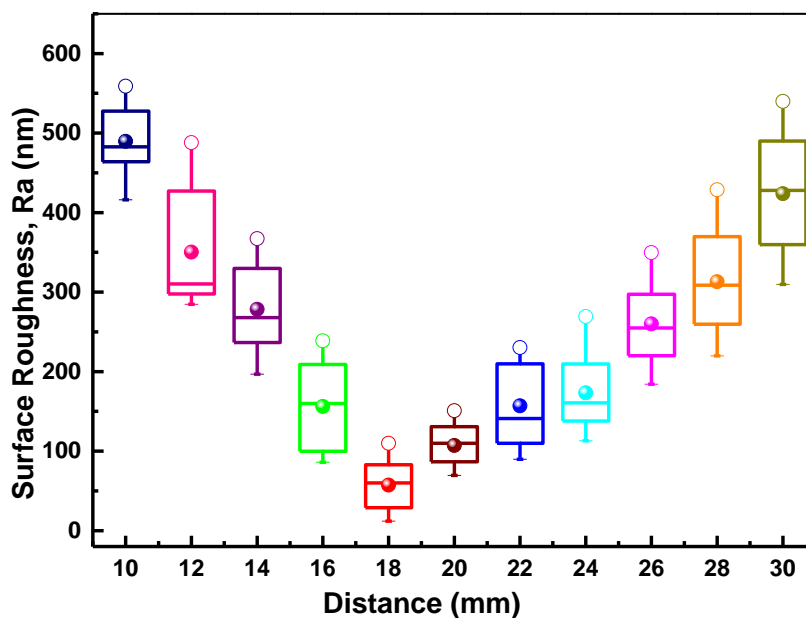


Figure 6. Box plot of stent surface roughness with different distances between electrodes (10–30 mm)

According to the conclusions in Table 4, the distance between the poles is the third factor that affects the quality of the stent. Therefore, a single-factor test is required for the distance between the poles. The experiment uses the distance between the poles as a single variable, with a value range of 10–30 mm. According to the conclusions in Sections 3.2.1 and 3.2.2, the best polishing time is 11 min, and

the best voltage for polishing is 24 V. The electrolyte temperature is tentatively set at 20 °C according to the orthogonal optimization parameters obtained in Tables 3 and 4. After the experiment, the box plot of the influence of different distances between electrodes on the surface roughness is shown in Figure 6.

Figure 6 shows that with the gradual increase in the distance between the cathode and anode, the surface roughness of the nitinol stent also shows a change law that initially decreases and then increases. When the distance between the electrodes is 10 mm, the minimum, maximum, and average surface roughness of the stent are 416.12, 558.98, and 489.58 nm, respectively. The roughness of stent gradually decreases with the increase in the distance between the poles. When the distance is 18 mm, the minimum surface roughness is obtained. At this time, the corresponding minimum, maximum, and average values are 11.87, 109.83, and 57.19 nm, respectively. When the distance exceeds 18 mm, the surface quality of the stent begins to gradually decrease. Especially when the distance is 30 mm, the minimum, maximum, and average surface roughness of the stent are 309.71, 539.85, and 423.87 nm, respectively. Compared with the best distance (18 mm), the surface quality has dropped remarkably.

3.2.4 Optimization of electrolyte temperature

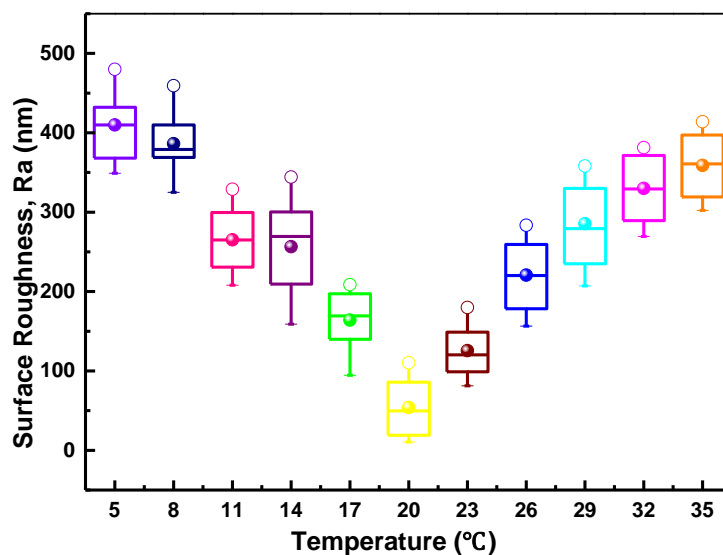


Figure 7. Box plot of surface roughness values for different electrolyte temperatures (5 °C–35 °C)

The electrolyte temperature has the least influence on the surface quality of the stent. Therefore, the single-factor test is placed last. Through the temperature control function of the magnetic stirrer, the temperature of the electrolytic polishing solution is changed, and the temperature range is 5 °C–35 °C. According to the conclusions of the first three sections, the best polishing time is 11 min, the voltage for polishing is 24 V, and the distance between electrodes is 18 mm. After the experiment, the corresponding surface roughness change box plot is shown in Figure 7.

Fig. 7 shows that the surface roughness of the stent initially decreases and then increases as the temperature of the electrolyte increases. When the electrolyte temperature is 5 °C, the minimum surface

roughness of the stent is 348.93 nm, the maximum roughness is 479.91 nm, and the average roughness is 409.84 nm. As the temperature of the electrolyte increases, the minimum, maximum, and average values of roughness gradually decrease. When the electrolyte temperature is 20 °C, the minimum, maximum, and average surface roughness of the stent are 10.59, 110.27, and 53.92 nm, respectively. Compared with the roughness when the electrolyte temperature is 5 °C, the roughness is reduced by 96.97%, 77.02%, and 86.84%. At this time, the surface roughness of the stent is minimum. The roughness gradually starts to increase as the temperature of the electrolyte continues to increase. When the temperature of the electrolyte is 35 °C, the minimum, maximum, and average roughness are 302.23, 413.98, and 358.85 nm, respectively. Compared with the best electrolyte temperature, it is increased by 27.54, 2.75, and 5.66 time. Therefore, the best electrolyte temperature is 20 °C.

3.3 Nitinol removal rate and surface morphology

3.3.1 Bridge width and wall thickness removal rate

As a medical device, high-quality nitinol stents are required for implantation in the human body [30]. Therefore, the dimensional changes and surface quality of the stent after electrolytic polishing should be studied accordingly. The variation of the width and wall thickness of the support bridge with polishing time is investigated on the basis of the optimized parameters in Sections 3.1 and 3.2. The optical profiler is used to perform noncontact measurement to ensure the accuracy of the corresponding size and avoid the deformation of the stent due to the stress caused by the contact measurement. The change curve of bridge width and wall thickness with polishing time is shown in Fig. 8. The figure shows that the bridge width and wall thickness are correspondingly reduced and generally appear linear as the polishing time is prolonged. When the polishing time is 11 min, the wall thickness is reduced by 36.47 μm , and the material removal rate is 3.32 $\mu\text{m}/\text{min}$. The bridge width is reduced by 39.37 μm , and the material removal rate is 3.58 $\mu\text{m}/\text{min}$. The width of the bridge is removed faster. On the one hand, this condition may be because the burrs, recast layers, and heat-affected zones produced by laser cutting are easier to remove [31]. On the other hand, this finding may be because the cutting surface receives a larger area covered by the electric field, resulting in a more intense electrolysis reaction [32]. The removal of wall thickness and bridge width is greater than 25 μm . Thus, the recast layer and slag on the stent can be completely removed.

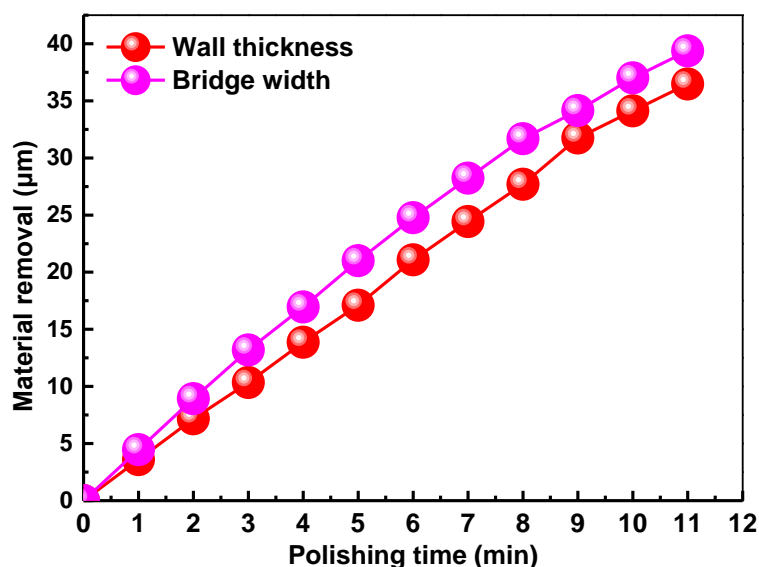


Figure 8. Changes in bridge width and wall thickness of the stent during different polishing time (0–11 min)

3.3.2 Polishing effect before and after electrolytic polishing

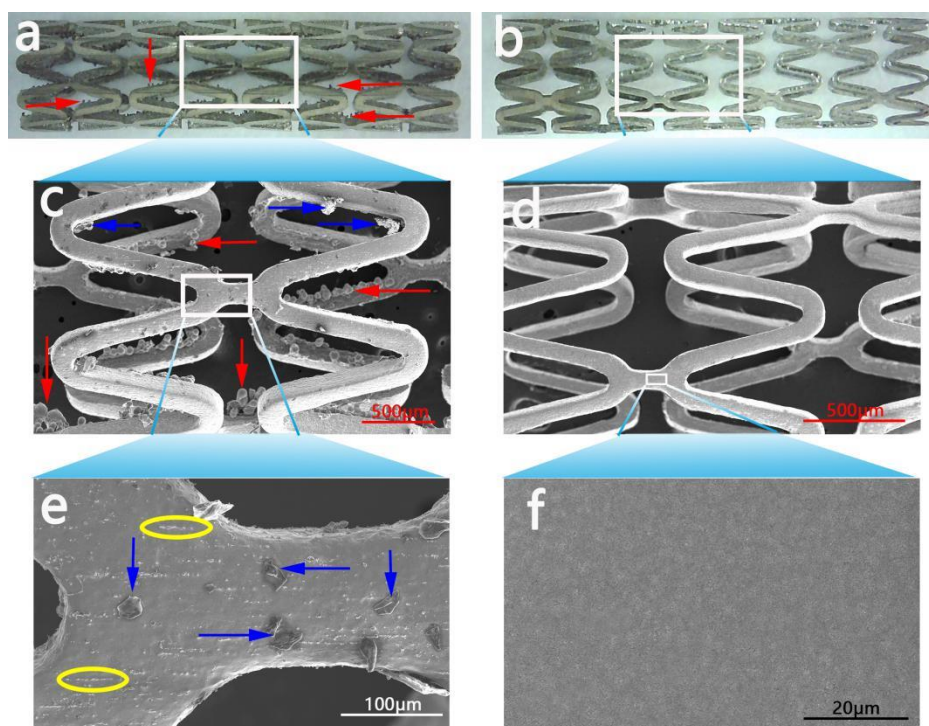


Figure 9. Surface visual image and micro morphology of the nitinol stent: before electrochemical polishing (a) (c) (e), after electrochemical polishing (b) (d) (f)

Figure 9 shows a comparison diagram of nitinol stent before and after electrolytic polishing. Figures 9(a), 9(c), and 9(e) show the low-, medium-, and high-magnification images of the stent prior to

polishing, respectively. Figures 9(a) and 9(c) clearly show the large amount of slag on the surface of the stent. After further magnification, Figure 9(e) shows that the microscopic surface of the stent is uneven and the flatness is poor. Figures 9(b), 9(d), and 9(f) are the low, medium, and high magnification images of the stent after polishing. The figures show that after electrolytic polishing, the slag adhering to the stent has been completely removed. The high-magnification image of Figure 9(f) shows that the surface of the stent is flat, no bumps and pits exist, and the microsurface quality has been greatly improved.

3.4 Comparison with similar results

In a previous study [24], nitinol tube was used as the research object in the case of perchloric acid, and methanol was the main electrolyte. The effect of polishing parameters on the surface morphology was studied. The best combination of electrolytic polishing process parameters was determined. The abovementioned previous study [24] was one of the foundations of the current paper, in which the electrolytic polishing solution was further optimized, and an environmentally friendly electrolytic polishing solution was used to avoid the damage of the acid electrolyte to the environment. In addition, the research objects also differed. In the current paper, the nitinol stent with a complex shape was the research object. The material removal rate of the stent wall thickness and bridge width were studied. In other studies [33][34][35], nitinol plates were used as the research object; the electrolytic polishing in acidic electrolytes, such as sulfuric, acetic, and phosphoric acids, were studied; and the surface morphology was optimized. Many scholars also pursued this research direction. Most scholars did not conduct a separate study on the influencing factors of additives and electrolytic polishing and did not consider the material removal rate. The research object and electrolyte of a previous study [25] were consistent with those in the current paper, but their polishing efficiency was low (20 min), and the influencing factors and material removal rate of electrolytic polishing were not studied. The average surface roughness in this article is relatively small, which reduced the adverse effects of the stent on the human body [36,37]. See Table 5 for specific comparison.

Table 5. Compared with similar discussion and results

Ref.	Research object	Electrolyte	Additive experiment	Factor influence (decreasing)	Average Ra (nm)	Material removal rate
[24]	Nitinol tube	perchloric acid and methanol	triethanola mine and absolute ethanol	exist	48.6	N.A.
[33]	Nitinol plate	sulfuric acid and methanol	N.A.	N.A.	32	N.A.

[34]	Nitinol plate	acetic acid	N.A.	N.A.	Increase 0.43	N.A.
[35]	Nitinol plate	phosphoric acid, sulfuric acid and distilled water	N.A.	N.A.	41.3	N.A.
[25]	Nitinol stents	sodium chloride-ethylene glycol and ethanol	N.A.	N.A.	N.A.	N.A.
This work	Nitinol stents	sodium chloride-ethylene glycol and ethanol	saturated potassium chloride solution	polishing time, voltage for polishing, distance between electrodes, polishing temperature	10.6	wall thickness: 3.32 $\mu\text{m}/\text{min}$; bridge width: 3.58 $\mu\text{m}/\text{min}$

4. CONCLUSIONS

For the first time, nitinol stent with complex shape is considered the research object, and the use of optimized environmentally friendly electrolytic polishing solution for electrolytic polishing is reported. The importance of electrolytic polishing factors is studied, further parameter optimization is performed, and the best electrolytic polishing parameters are obtained. After polishing, the surface quality of the stent has been greatly improved. The main conclusions are as follows:

(1) An amount of 23 ml/L of saturated potassium chloride solution is added to 30% volume fraction of absolute ethanol and 1 mol/L sodium chloride-glycol electrolyte to obtain the best environmentally friendly electrolytic polishing solution.

(2) In the polishing process of environmentally friendly electrolyte, the degree of influence on the electrolytic polishing effect in descending order is arranged as follows: polishing time, polishing voltage, distance between electrodes, and electrolyte temperature. The best polishing parameters are polishing time 11 min, polishing voltage 24 V, distance between electrodes 18 mm, and electrolyte temperature 20 °C.

(3) Under these electrolyte and optimal polishing parameters, the wall thickness of the stent is reduced by 36.47 μm , and the material removal rate is 3.32 $\mu\text{m}/\text{min}$. The bridge width is reduced by 39.37 μm , and the material removal rate is 3.58 $\mu\text{m}/\text{min}$. The recast layer and slag on the stent are completely removed, and the surface quality is improved. The minimum, maximum, and average surface roughness of the stent are 10.59, 110.27, and 53.92 nm, respectively.

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References

1. M.I. Hervella, C.C. Munuera, D.O. Beltrán, A.L. Pineda, V.B. González, V.F.G. Guillén, R. Pascual and J.A. Quesada, *Rev Esp Cardiol.*, 2 (2020) 125. <https://doi.org/10.1016/j.recesp.2020.09.012>
2. H. Zhou, S. Wang, L. Seidlein and X. Wang, *Front. Med.* 14 (2020) 1. <https://doi.org/10.1007/s11684-019-0733-5>
3. D. Aune, E. Giovannucci, P. Boffetta, L.T. Fadnes, N. Keum, T. Norat, D.C. Greenwood, E. Riboli, L.J. Vatten and S. Tonstad. *Int. J. Epidemiol.* 46 (2017) 1029. <https://doi.org/10.1093/ije/dyw319>
4. B. O'Brien and W. Carroll, *Acta Biomater.*, 5 (2009) 945. <https://doi.org/10.1016/j.actbio.2008.11.012>
5. E.Y. Chow, Y. Ouyang, B. Beier, W.J. Chappell and P.P. Irazoqui, *IEEE Trans. Microwave Theory Tech.*, 57 (2009) 2523. <https://doi.org/10.1109/TMTT.2009.2029954>
6. E. Pujiyulianto, Y. Amalia, T. Wahyuningsih, Y.P.G. Frideni, Field and R. Z. Mirahati, *Key Eng. Mater.*, 867 (2020) 1. <https://doi.org/10.4028/www.scientific.net/KEM.867.1>
7. E. Pujiyulianto and Suyitno. *INT. J. ADV. MANUF. TECH.*, 112 (2021) 1. <https://doi.org/10.1007/s00170-020-06484-3>
8. M. Bhaumik and K. Maity, *Surf. Rev. Lett.*, 24. (2017) 2. <https://doi.org/10.1142/S0218625X18500294>
9. T. Duerig, A. Pelton and D. Stöckel, *Mater. Sci. Eng., A*, 273 (1999) 149. [https://doi.org/10.1016/S0921-5093\(99\)00294-4](https://doi.org/10.1016/S0921-5093(99)00294-4)
10. M. Schillinger, S. Sabeti and C. Loewe, *J. VASC. SURG.*, 44 (2006) 684. <https://doi.org/10.1016/j.jvs.2006.07.008>
11. K. Weinert, V. Petzoldt and D. Kötter, *CIRP Ann.*, 53 (2004) 65. [https://doi.org/10.1016/S0007-8506\(07\)60646-5](https://doi.org/10.1016/S0007-8506(07)60646-5)
12. T. Ezaz, J. Wang, H. Sehitoglu and H.J. Maier, *Acta Mater.*, 61 (2013) 67. <https://doi.org/10.1016/j.actamat.2012.09.023>
13. Y. Zhong, K. Gall and T. Zhu, *Acta Mater.*, 60 (2012) 6301. <https://doi.org/10.1016/j.actamat.2012.08.004>
14. S.H. Mills, C. Dellacorte, R.D. Noebe, B.Amin-Ahmadi and A.P. Stebner, *Acta Mater.*, 209 (2021) 116784. <https://doi.org/10.1016/j.actamat.2021.116784>
15. D. Herzog, P. Jaeschke, O. Meier and H. Haferkamp, *Int. J. Mach. Tool. Manu.*, 48 (2008) 1464. <https://doi.org/10.1016/j.ijmachtools.2008.04.007>
16. R. Siebert, J. Schneider and E. Beyer, *IEEE Trans. Magn.*, 50 (2014) 1. <https://doi.org/10.1109/TMAG.2013.2285256>
17. R. Akarapu, B.Q. Li and A. Segall, *J. Fail. Anal. Prev.*, 4 (2004) 51. <https://doi.org/10.1361/15477020420756>
18. S.H. Lee and S.E. Ahn. *JSME Int J., Ser. C*, 19 (2003) 1591. <https://doi.org/10.1299/jsmec.46.1591>
19. E. Pujiyulianto and Dr. Suyitno. *Adv. Mater. Res.*, 1154, (2019) 91. <https://doi.org/10.4028/www.scientific.net/AMR.1154.91>
20. T.H. Shin, S.Y. Baek and E.S. Lee. *Adv. Mater. Res.*, 79 (2009) 155. <https://doi.org/10.4028/www.scientific.net/AMR.79-82.155>
21. E.S. Lee and T.H. Shin, *J. Mech. Sci. Technol.*, 25 (2011) 963. <https://doi.org/10.1007/s12206->

011-0209-2

22. N.I. A. Lopes, L.O. Silva, L.A. Santos and V.T.L. Buono, *Mater. Res.*, 20 (2017) 572.
<https://doi.org/10.1590/1980-5373-mr-2016-0933>
23. D. Han, H. Yang, M.S. Kong, C. Lee, A. Sharma and B. Ahn, *Mater. Express*, 10 (2020) 1249.
<https://doi.org/10.1166/mex.2020.1744>
24. H. Ji, Y. Wang, Z. Li, Z. Huang and M. Chai, *Int. J. Electrochem. Sci.*, 16 (2021) 210364.
<https://doi.org/10.20964/2021.03.42>
25. Y.Q. Wang, X.T. Wei and Z.Y. Li, *Int. J. Electrochem. Sci.*, 15 (2020) 8823.
<https://doi.org/10.20964/2020.09.01>
26. X. Wu and D.Y.C. Leung, *Appl. Energy*, 88 (2011) 3615.
<https://doi.org/10.1016/j.apenergy.2011.04.041>
27. L. Deng, B. Feng and Y. Zhang, *Ceram. Int.*, 44 (2018) 15918.
<https://doi.org/10.1016/j.ceramint.2018.06.010>
28. C.C. Zhou, G.F. Yin and X.B. Hu, *Mater. Des.*, 30 (2009) 1209.
<https://doi.org/10.1016/j.matdes.2008.06.006>
29. E.S. Lee, *Int. J. Adv. Manuf. Technol.*, 16 (2000) 591. <https://doi.org/10.1007/s001700070049>
30. W. Haider, N. Munroe, C. Pulletikurthi, P.K.S. Gill and S. Amruthaluri, *J. Mater. Eng. Perform.*, 18 (2009) 760. <https://doi.org/10.1007/s11665-009-9435-5>
31. Á. Lengyel, P. Nagy, E. Bognár and K. Hirschberg, *Mater. Sci. Forum*, 729 (2012) 436.
<https://doi.org/10.4028/www.scientific.net/MSF.729.436>
32. D.R. Turner, *J. Electrochem. Soc.*, 105 (1958) 402. <https://doi.org/10.1149/1.2428873>
33. K. Fushimi, M. Stratmann and A.W. Hassel, *Electrochim. Acta*, 52 (2006) 1290.
<https://doi.org/10.1016/j.electacta.2006.07.030>
34. P. Shi, F.T. Cheng and H.C. Man, *Mater. Lett.*, 61 (2007) 2385.
<https://doi.org/10.1016/j.matlet.2006.09.020>
35. L. Neelakantan and A. WalterHassel, *Electrochim. Acta*, 53 (2007) 915.
<https://doi.org/10.1016/j.electacta.2007.08.007>