Study on electrochemical polishing of TC4 alloy

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Abstract

Using an environmentally friendly acid-alcohol system solution for electrochemical polishing of 3D printed titanium alloy, a typical U-I polarization curve was obtained, and explained the mechanism of electrochemical polishing. In this paper, the influence of electrochemical polishing factors (electrolyte temperature, polishing distance, and polishing time) on the surface roughness of Ti-6Al-4V (TC4) was studied, and optimized the process parameters to obtain a surface roughness of 0.3 \( \mu \text{m} \) (1 mm \( \times \) 1 mm). Electrochemical polishing can reach the level of mechanical grinding and polishing, meeting the requirements for subsequent assembly and use.

Introduction

Ti-6Al-4V (TC4) alloys are widely used in aerospace, military, and civil industries because of their good comprehensive mechanical properties, high specific strength, corrosion resistance, high - temperature resistance, and low thermal conductivity [1–5]. Their use accounts for 75%–85% of all titanium alloys. However, untreated TC4 has a large surface roughness, which seriously affects its performance [6–9]. Electrochemical polishing is an important surface treatment technology for metal materials. It can not only reduce the surface roughness of the material, but also effectively improve the brightness and pollution resistance [10–12]. Therefore, the surface polishing of TC4 alloy by electrochemical polishing is the key to its wide application [13–17].

Bagehorn et al [18] developed an enhanced electrochemical polishing process that reduced the surface roughness of complex-shaped Ti-6Al-4V parts by approximately 84% and improved fatigue performance. Bao Shenghua et al [19] introduced power ultrasonication in the process of electropolishing Ti-6Al-4V and analyzed the effect of power ultrasonication on electrolytic polishing. Xu Xiaojing et al [20] performed electrochemical polishing and anodizing treatments on TC4 titanium alloy and studied the morphology and phase of the treated surface. Fan Lixia et al [21] used 5% perchloric acid ethanol solution to electropolish TC4 titanium alloy, measured the corresponding electropolishing characteristic curve, and obtained the best electropolishing conditions 0... for the alloy: an electropolishing temperature of 20 °C, the voltage of 28 V, the current density of 0.98 A cm\(^{-2}\), puffing, and electrolytic polishing time of 25 s.

In this study, a low-cost and easy-to-operate electrochemical polishing technology is adopted to improve the surface quality of 3D printed titanium alloy parts after abrasive flow processing. The effects of electrochemical polishing factors (current and voltage, electrolyte temperature, polishing time, and polishing distance) on the surface roughness of Ti-6Al-4V (TC4) were studied by selecting a polishing solution consisting of an acid-alcohol system, optimized to determine the best parameters for each influencing factor, and the performance of the polished samples was tested and analyzed.

Experiment

The process of using electrochemical polishing technology to polish the surface of 3D printed titanium alloys includes cleaning titanium alloy samples, preparing polishing solutions, and electrochemical polishing. The
main parameters to be considered in the optimization process are the current and voltage, electrolyte temperature, polishing time, and polishing distance. The experimental process is illustrated in figure 1.

During the electrochemical polishing process, TC4 alloy was used as the anode (held by a clamp), the cathode plate was made of 304 nickel-based stainless steel, and the surface area ratio of the positive electrode to the cathode was maintained at 1:9. The size of the TC4 sample used in the experiment was 10 × 10 × 5 mm. Before the experiment, the samples and plates were ultrasonically cleaned with deionized water, acetone, and anhydrous ethanol at room temperature for 10 min. To remove the oxide layer from the surface, the TC4 sample was placed in an acidic solution for 30 s. Finally, anhydrous ethanol and deionized water were used for ultrasonic treatment for 10 min.

The experiment used perchloric acid-methanol system electrolyte to polish 3D printed titanium alloy [22–24]. Its composition was: perchloric acid (HClO₄, 70%–72%, density 1.76 g cm⁻³), accounting for 8%; methanol (CH₃OH, 99.5%, density 0.79 g cm⁻³), accounting for 75%; ethylene glycol ((CH₂OH)₂, 99%, density 1.11 g cm⁻³), accounting for 17%.

First, the voltage and current are adjusted to obtain a dynamic U-I curve. According to the U-I curve, the polarization behavior of TC4 alloy sample in polishing solution is studied and determined the optimal voltage and current in the polishing process. Then, the effect of polishing solution temperature (set at 15, 20, 25, 30, 35, and 40 °C) on TC4 polishing effect was studied by fixed voltage, current, plate spacing, and polishing time; Also, the effects of plate spacing (2, 3, 4, 5 cm) on the polishing effect were studied by fixing the voltage, current, polishing solution temperature and polishing time; Finally, the voltage and current, electrolyte temperature and the distance between anode and anode plate were determined, and the influence of polishing time (set to 2, 4, 6, 8 and 10 min) on polishing effect was studied.

The surface roughness was measured by Bruker Icon type atomic force microscope. The three-dimensional microstructures were analyzed by Laser scanning confocal microscope. The morphology and elements of the samples before and after polishing were analyzed by Quanta 450 field emission scanning electron microscope. The phase analysis of the surface before and after polishing was carried out by D8 advanced x-ray diffractometer.

Results and discussion

U-I curve

Figure 2 shows the polarization curve of 3D printed TC4 alloy. As shown in the figure, the U-I curve of the electrochemical polishing of TC4 alloy presents a typical metal electrochemical polishing curve, which is divided into three stages [25]. When the current density of the electrode is low, no electrochemical reaction occurs. When the current density further increases, the surface of the TC4 alloy begins to undergo a reaction. The metal atom loses electrons and becomes metal ions in the solution, as follows:

\[ M = M^{n+} + n^e^- \]  \hspace{1cm} (1)

\[ M^{n+} + \chi H_2O = M^{n+} \cdot \chi H_2O \]  \hspace{1cm} (2)

At a suitable value, a passivation film forms on the surface of the alloy, and a typical passivation reaction occurs. The metal atoms on the surface of the anode base substrate lose electrons and undergo oxidation reaction, resulting in a low-valence oxide:

\[ M + 2OH^- = MO + H_2O + 2e^- \]  \hspace{1cm} (3)

\[ M + 4H_2O = M_2O_4 + 8H^+ + 8e^- \]  \hspace{1cm} (4)
At the same time, the oxides formed on the surface of the TC4 sample begin to become metal ions in the polishing solution, and the cathode surface begins to undergo a reduction reaction to generate oxygen:

\[ MO + 2H^+ = M^{2+} + H_2O \]  

\[ 4OH^- = O_2 + H_2O + 4e^- \]

In summary, when the voltage is about 20 V, the current density is about 0.339 A cm\(^{-2}\), electrochemical polishing has the best effect.

The surface micro-morphology of TC4 alloy under different current densities is shown in figure 3 (the size range of all three-dimensional micro-profile topography images is 1280 \( \mu \)m \( \times \) 1280 \( \mu \)m). It can be observed from the figure that the surface is relatively rough when the current density is 0.137 A cm\(^{-2}\). Although some pits on the original surface were improved, they are still visible. When the current density increased to 0.293 A cm\(^{-2}\), the original pits were eliminated and the surface became smoother, but there were still some fluctuations in morphology. When the current density was 0.339 A cm\(^{-2}\), the remaining fluctuation disappeared, and the surface became smooth and bright. When the current density was increased to 0.74 A cm\(^{-2}\), the solution boiled violently during polishing and produced a large number of bubbles. As the solution became turbid, the microcosmic surface of the sample increased in brightness, but black pits and pits appeared with uneven distribution. This may be because the current density is too large, the metal ions on the surface of the anode sample dissolve too quickly, and the large amount of gas generated during the polishing process acts as a stirring effect, further accelerating the dissolution of the anode metal, making polishing effects such as over-polishing and corrosion difficult to control, which greatly affects the surface quality.

Figure 4 shows the variation of the TC4 alloy surface roughness Ra value with current density. As can be seen from the figure, during the electrochemical polishing process, with increasing current density, the Ra value on the surface of the TC4 alloy sample first decreases and then increases. When the polishing current density was low, the metal on the surface of the sample dissolved slowly, and the polishing efficiency was also low, so that only part of the surface scratches and pits were removed. With an increase in the current density, the dissolution rate of metal ions on the surface of the anode sample accelerated, and the scratches and pits were gradually eliminated. When the current density was 0.339 A cm\(^{-2}\), the surface of the sample was the smoothest and brightest, and the surface roughness Ra also dropped to 0.81 \( \mu \)m (1.28 mm \( \times \) 1.28 mm). As the current density continued to increase, the polishing liquid boiling phenomenon occurred, accompanied by a large amount of gas generation, resulting in excessive polishing, pitting, and pits on the surface of the sample, which seriously affected the surface quality. Therefore, according to the above analysis, 0.339 A cm\(^{-2}\) is the optimal current density for electrochemical polishing of the 3D-printed TC4 titanium alloy, and the voltage value at this time was 20 V.

**Electrolyte temperature**

To determine the influence of the temperature of the electrolytic polishing solution on the polishing quality of the 3D printed TC4 alloy surface, the other polishing parameters were fixed as follows: a polishing voltage of 20 V, electrode spacing of 4 cm, polishing time of 4 min, and the constant temperature of the polishing solution was set at 15, 20, 25, 30, 35, and 40 °C for the electrochemical polishing of the 3D printing TC4 alloy.
Figure 5 shows the 3D microprofile topography of the TC4 alloy surface after electrochemical polishing at different temperatures. As can be seen from figure 5(a), when the constant temperature of the polishing solution was 15 °C, there were still a large number of pits on the surface of the polished TC4 alloy sample, and several pits indicated that the electrochemical polishing effect was not good when the solution temperature was too low. When the solution temperature remained constant at 20 °C, most of the polished surface of the TC4 alloy was relatively flat, without obvious pits and only a few over-corrosion points. The surface was relatively smooth and
bright, and the polishing effect was good, as shown in figure 5(b). When the temperature of the polishing solution increased to 25 °C, there were no obvious pits on the surface of the titanium alloy sample, but a large number of over-corroded areas appeared, as shown in figure 5(c). As the temperature continued to rise, a large number of over-corroded areas and spots continued to appear on the polished surface of the TC4 titanium alloy, and uneven pits and bumps appeared on the surface. This shows that as the temperature of the polishing solution increases, the viscosity of the solution is greatly reduced, and the diffusion ability of free ions in the solution becomes stronger, resulting in a faster dissolution rate for the metal ions on the surface of the titanium alloy, resulting in a large number of over-corroded areas and ripples, which can greatly affect the surface quality of the sample.

Figure 6 shows the variation trend curve of the surface roughness Ra value of 3D printed TC4 alloy with polishing temperature after polishing. It can be clearly seen from the figure that, as a whole, the surface roughness Ra value of the sample first decreases and then increases with increasing temperature of the polishing liquid. When the polishing liquid temperature was 15 °C, the low polishing liquid temperature led to high viscosity of the solution and weak ion diffusion ability, resulting in a rough surface of the sample. As the temperature of the solution increased, the viscosity of the solution gradually increased. As the amount of solution decreases and the solution is activated, the surface roughness of the TC4 alloy gradually decreases as the polishing progresses, and the surface quality of the sample is improved. It can be seen from the figure that the lowest surface roughness for the TC4 alloy is at a polishing temperature of 20 °C, where the Ra is 0.411 μm (1.28 mm × 1.28 mm). At this point, the surface of the sample was relatively flat. Then, as the polishing fluid temperature continued to increase, the surface roughness Ra of the sample increased. There are many over-corroded areas and pits on the surface of the TC4 alloy sample. From the above analysis, it can be concluded that the polishing temperature affects the activity and viscosity of the solution. When the temperature was low, the activity of the polishing liquid was low, the viscosity was large, the diffusion ability of ions in the solution was weak, the polishing effect of the sample surface was relatively poor, and the improvement of the surface quality was not obvious. When the polishing temperature is too high, the solution has high activity and low viscosity, which leads to strong diffusion ability of ions in the polishing solution and violent reaction on the sample surface; thus, it easily adheres to the sample. There is a large amount of over-corrosion on the surface, which greatly affects the polishing quality of the sample surface. In summary, the polishing temperature should be 20 °C to obtain samples with the best polishing quality.
Polishing pole distance

To explore the effect of electrode plate spacing on the surface polishing quality of 3D printed TC4 alloy during electrochemical polishing, other polishing parameters were fixed as follows: a polishing voltage of 20 V, polishing temperature of 20 °C, polishing time of 4 min, and electrode spacing set to 2, 3, 4, and 5 cm for the electrochemical polishing of 3D printing TC4 alloy.

Figure 7 shows the 3D micro-profile topography of the TC4 alloy surface after electrochemical polishing at different electrode spacing: (a) 2 cm, (b) 3 cm, (c) 4 cm, (d) 5 cm.

Figure 6. Curve of values of the surface roughness with polishing solution temperature.
large number of bubbles appeared in the anode solution when the electrode spacing was 2 cm. The temperature measured by the thermometer showed that the local temperature was higher, which resulted in a faster metal dissolution rate on the surface of the sample and a large number of defects such as ripples and pits. As the distance between the electrodes increased to 3 cm, as shown in figure 6(b), the polished sample surface still had pits, ripples, over-corrosion, and other defects, but the number was significantly reduced, and the polishing effect was improved. When the electrode spacing was 4 cm, the sample surface was relatively flat, without obvious pits and ripples, and with few defects, as shown in figure 6(c). When the distance between the poles was 5 cm, there were over-eroded and polished areas and a few pits on the surface, and the polishing effect became worse, as shown in figure 6(d). This is because at the same voltage, the distance between the electrodes increases and the current density decreases, resulting in less energy in the polishing process and a weaker polishing effect.

Figure 8 shows the variation curve of the surface roughness Ra of the TC4 alloy with the distance between the plates. It can be seen from the figure that the Ra value first decreases and then increases with increasing the electrode plate spacing. When the distance between the electrodes is small, the diffusion of dissolved ions in the area near the anode in the polishing solution is blocked, and the polishing reaction is intense, resulting in a large number of ripples, pits, and other defects on the surface, resulting in poor surface quality. As the distance between the electrodes increased, the polishing reaction tended to be steady and smooth, and the polishing effect improved. When the distance between the electrodes was 4 cm, the Ra value of the TC4 alloy sample was the smallest at 0.365 μm (1.28 mm × 1.28). At this time, the surface was relatively flat and smooth. After that, the distance between the poles continued to increase, pits appeared on the surface, the polishing effect became worse, the Ra value began to increase, and the surface quality of the sample deteriorated. Based on the above analysis, it was determined that the best plate spacing for electrochemical polishing of 3D printing TC4 alloy was 4 cm.

Polishing time
To study the effect of the electrochemical polishing time on the surface polishing quality of 3D printed TC4 alloy samples, the polishing voltage was set to 20 V, the polishing temperature was 20 °C, the distance between the anode and the cathode was 4 cm, and the polishing time was set to 2, 4, 6, 8, and 10 min to explore the influence of different electrochemical polishing times on the surface of the sample.

Figure 9 shows the 3D micro-profile topography of the TC4 alloy surface after electrochemical polishing at different polishing times. It can be seen from figure 9(a) that when the polishing time was 2 min, the polishing time was too short and the polishing process was not completed. There are still defects such as incompletely dissolved bumps and pits on the surface, and the polishing effect is poor. With the increase of time, the surface gradually flattens out, and surface defects such as pits are reduced. When the polishing time was 6 min, the polishing effect of the sample surface was good, smooth, and bright, without obvious defects, and good surface quality, as shown in figure 9(c). Subsequently, the polishing time continued to increase. Although the surface of the sample is relatively flat, there are differences in brightness and darkness, as well as corroded black areas, as shown in figure 9(d). This is because the polishing time is prolonged and the surface of the sample is severely

![Figure 8. Curve of values of the surface roughness with the distance between plates.](image-url)
corroded, and as the polishing time continues to increase, the entire surface of the sample becomes darker and the surface quality becomes worse, as shown in figure 9(e). The polished sample was over-polished, and the shape of the sample changed significantly.

Figure 10 shows the variation curve of the TC4 alloy surface roughness with different polishing times. It can be seen from the figure that as the polishing time increased, the sample surface roughness value first decreased and then increased. When the polishing time was 2 min, the time was shorter, the metal polishing on the sample surface was not completely dissolved, and the roughness Ra value was large. When the polishing time was 6 min, the sample was polished and dissolved in the polishing solution for a suitable time, and the sample surface was smooth and bright with low roughness. When the polishing time was increased to 10 min, the sample was over-polished in the polishing liquid, and the surface roughness increased because of over-corrosion and polishing. Therefore, the best polishing time for the electrochemical polishing of 3D printed TC4 alloy samples is 6 min.

**Uniformity analysis**

Figure 11 shows the EDS analysis diagram of the sample surface and the yellow substance before polishing. As shown in the figure, the main components of the surface before polishing were Al, Ti, and V. The main
Figure 10. Curve of values of the surface roughness with different polishing time.

Figure 11. The EDS spectrum of TC4 alloy surface: (a) before polishing; (b) not cleaned after polishing.

Figure 12. XRD patterns of TC4 Alloy under different conditions: (a) not cleaned after polishing; (b) cleaned after polishing; (c) before polishing.

\[
\begin{align*}
Ti^{4+} + Cl^- & \rightarrow TiCl_4 \\
TiCl_4 + 2H_2O & \rightarrow TiO_2(s) + 4H^+ + 4Cl^- 
\end{align*}
\]
components of the yellow substance are Al, Ti, V, Cl, O, and other elements. The atomic ratios of Al and Cl are close to 1:4, and the ratios of Ti and O are close to 1:2, which may be AlCl$_4$ and TiO$_2$, respectively. Through x-ray diffraction test analysis of the samples before polishing, uncleaned after polishing, and cleaned after polishing, the yellow substance was found to be a mixture of AlCl$_4$ and TiO$_2$, as shown in figure 11. This is because during the polishing process, the thickness of the passivation film formed by the convex particles and the concave part of the sample surface are different. The metal Ti in the convex part of the particles is first dissolved under the action of the current, and Ti$^{4+}$ diffuses to the passivation film. In solution, it reacts with Cl$^{-}$ in the solution to form titanium tetrachloride. The reaction process is shown in equation (7). Titanium tetrachloride is a high-viscosity yellow liquid that is difficult to separate from the surface of the sample to form a titanium tetrachloride layer; as the reaction progresses, titanium tetrachloride reacts with water molecules to form titanium dioxide. The reaction process is shown in equation (8). Titanium dioxide has strong adhesion to metal titanium, so titanium dioxide adheres to the surface of the sample to form a stable. In this way, a dynamic reaction process is formed, which gradually dissolves and reacts on the convex part of the metal sample surface, and finally makes the metal surface smooth, achieving a leveling effect.

Figure 13. The curve of surface microhardness of TC4 alloy with polishing time.

Figure 14. Potentiodynamic polarization curves of TC4 alloy before and after electrochemical polishing.
Figure 12 shows the change curve of the surface microhardness of the TC4 alloy with polishing time. It can be seen from the figure that the surface microhardness of the polished sample is lower than that of the unpolished sample, which is approximately 4% lower than that of the unpolished sample. With an increase in the polishing time, the microhardness of the sample surface showed a decreasing trend. The change in the surface hardness of the sample before and after polishing was related to the surface hardening layer and residual stress. This is because the 3D printing TC4 alloy was fabricated by layer-by-layer printing with high-energy electron beam molten metal powder. This type of manufacturing method causes a large amount of metal powder on the surface to be cooled too fast and remain without being integrated into the matrix, causing the surface to shrink and form stress. Therefore, electrochemical polishing can erode the surface-hardened layer and cause residual stress on the sample surface to a certain extent, resulting in a decrease in the surface hardness of the sample.

Figure 13 shows the potential polarization curves of the TC4 alloy samples before and after electrochemical polishing. It can be clearly seen from the figure that the Todenka polarization curves before and after polishing exhibit similar trends, and obvious passivation behaviors have occurred. The difference is that the self-corrosion potential $E_{\text{corr}}$ is different. The self-corrosion potential $E_{\text{corr}}$ of the sample surface before polishing was approximately $-1.0139$ V, and the self-corrosion potential $E_{\text{corr}}$ after polishing was approximately $-0.9453$ V, so the self-corrosion potential reflected the stability of the sample surface. With higher $E_{\text{corr}}$, the stability of the surface and the corrosion resistance both increase. Therefore, the corrosion resistance of the sample surface after electrochemical polishing was improved to a certain extent. Therefore, electrochemical polishing can obtain a
smooth and dense TiO₂ oxide layer, which improves the corrosion resistance of the TC4 alloy. In addition, the surface of the sample after electrochemical polishing is flatter and smoother than the original surface because the rougher surface has lower pitting sensitivity and is more prone to corrosion behavior.

Figure 14 shows the variation in the friction coefficient of the TC4 alloy samples with friction time before and after electrochemical polishing. Generally, the change curve of the friction coefficient with friction time goes through three stages: the running stage, stable stage, and wear stage. It can be seen from the figure that the trend of friction coefficient of the samples before and after electrochemical polishing is the same over time, and both experience a long initial wear stage before reaching a stable wear state. It can also be seen that for the samples before polishing, a sudden rise occurs after 1.5 min of friction, which may be caused by the loosening of a large number of unmelted powder particles attached to the sample surface before polishing. The polished sample maintained a steady growth trend as the friction coefficient increased sharply from the beginning. After stabilization, the friction coefficient of the sample before electrochemical polishing was higher than that of the sample after polishing. The friction coefficient before electrochemical polishing was 0.4281, and the friction coefficient after polishing was 0.3917, as shown in figure 15. There was little difference in the friction coefficients between the two. The reason for the slightly larger coefficient of friction before polishing may be related to the large number of metal particles on the surface of the sample, which are looser and fall off in a small area.

To further explore the reasons for the change in the friction coefficient and friction mechanism, SEM tests were conducted on the friction test samples before and after electrochemical polishing, as shown in figure 16. It can be seen from the figure that after the friction and wear of the sample before polishing, the grinding ball has many grooves and shedding along the reciprocating sliding direction. The area also has a large number of smooth friction bands and a few furrows. The most obvious wear mechanisms here are adhesive wear and peeling wear, as shown in figures 16(a) and (b). The surface of the sample after electrochemical polishing was relatively smooth after the friction and wear tests, and there was no surface shedding or grooves. In contrast, the width of the surface wear marks after friction was relatively narrow, and there were many deep grooves and scratches (furrows), where the wear mechanism is abrasive wear, as shown in figures 16(c) and (d).
Conclusion

An environmentally friendly polishing fluid (perchloric acid–methanol system) was used to obtain the best process parameters for electrochemical polishing: a polishing voltage of 20 V, a polishing current of 0.339 A cm⁻², an optimal polishing temperature of 20 °C, an electrode spacing of 4 cm, and a polishing time of 6 min. The surface roughness of TC4 alloy decreased to 0.3 μm (1 mm × 1 mm), which greatly improves the surface quality and provides an experimental basis for realizing the flattening of TC4 alloy surface.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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