Spray electrolyte plasma polishing of GH3536 superalloy manufactured by selective laser melting

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Abstract
Electrolyte plasma polishing (EPP) is an innovative and efficient technology mainly used to polish complex metal products and obtain surfaces with low roughness and high gloss. However, limited by the electrolyte bath size, EPP method is not suitable for polishing workpieces with large size. This paper introduces a spray electrolyte plasma polishing (SEPP) method that can process large-scale and complex surface. The theoretical mechanism of SEPP is presented. GH3536 superalloy plates manufactured by SLM were used as the experimental specimens. The influence of cathode feed velocity on surface roughness is studied. Key parameters, like distance between anode and cathode, voltage, temperature, and flow rate of the electrolyte were investigated experimentally. Properties such as surface roughness, micro morphology, chemical composition, and surface hardness of the GH3536 specimens were detected and analyzed. The results show that SEPP technology can decrease the specimen surface roughness from \( R_a = 13.93 \, \mu m \) to \( 0.107 \, \mu m \) and obtain a smooth and glossy surface without obvious machining traces. The oxides and other impurities on the specimen surface were removed, and the contents of Ni, Cr, and Fe elements were increased. Additionally, the surface hardness values of the polished specimen were increased and showed good consistency.

Keywords Spray electrolyte plasma polishing (SEPP) · Superalloy · Selective laser melting (SLM) · Surface roughness · Key parameters

1 Introduction
Additive manufacturing has shown extensive prospects in many aspects in the aerospace field, such as manufacturing of difficult-to-machine materials, integrated manufacturing of complex structures and functions, and manufacturing of lightweight structure [1]. However, defects such as staircase effect, balling effect, and powder adhesion in the additive manufacturing process will cause high roughness of the formed surfaces. So accordingly, additive manufactured parts generally need surface polishing to obtain high-quality surfaces and meet the high-service requirements.

Superalloy parts possess excellent high-temperature mechanical properties, oxidation resistance, and corrosion resistance. Additive manufactured superalloy parts are generally used in the key hot components of aero and rocket engine [2, 3]. To date, the main polishing methods for aerospace parts mainly include mechanical grinding [4], abrasive flow polishing [5, 6], and electrochemical polishing [7]. When machining complex superalloy parts, the above polishing methods can improve the surface quality significantly. However, problems such as surface damage, excessive surface residual stress, or environmental pollution are inevitable in the processing process [8]. Therefore, there is an urgent need to develop new techniques suitable for polishing complex superalloy parts.

EPP is an innovative and efficient technology mainly used to polish complex metal products and obtain surfaces with low roughness and high gloss. EPP is a composite process that removes protrusion materials of the metal surface through plasma-physical discharges and electrochemical reactions [9, 10]. EPP technology has recently drawn much attention due to its advantages such as high-quality surfaces, high efficiency, easy to polish complex workpieces, no tool contact, no additional residual stress,
and environment friendly. Some studies also show that surface properties such as wear resistance and corrosion resistance of EPP-treated workpiece are improved [11, 12]. EPP technology presents broad application prospects, which provides an opportunity to solve the finishing problem of complex additive manufactured superalloy parts. At present, most researchers are focusing on revealing the polishing mechanism and explore new polishable materials in the field of electrolyte plasma polishing [13–15]. The existing investigation results also suggest that EPP technology shows great application value in the medical field [16–18]. In 2021, Seo et al. [12] and Stepputat et al. [19] reported the significant polishing effect of EPP technology on additive manufactured parts. The application of EPP technology on the inner surface of metal pipes was achieved by Radkevich and Kuzmichev [20] and Cornelsen et al. [21]. However, limited by the size of the electrolyte bath and the current, the workpieces used in the above researches are generally small-sized parts; there is a lack of research on workpieces with large-size and complex shape.

In this paper, a spray electrolyte plasma polishing (SEPP) method for large-size and complex surfaces is proposed. The GH3536 superalloy plates produced by selective laser melting (SLM) were used as the experimental specimens. The influence of cathode feed velocity on surface roughness is studied. Key parameters, like distance between anode and cathode, voltage, temperature, and flow rate were investigated experimentally. Moreover, to demonstrate the polishing effect of SEPP technology, the surface roughness, micro morphology, chemical composition, and surface hardness of the specimen before and after polishing were detected and analyzed.

2 Methods and materials

2.1 Theoretical mechanism

Traditional EPP method polishes metal parts by completely soaking them into the electrolyte entirely, which is convenient and efficient in polishing small-size workpieces. The EPP process usually occurs at a high voltage between 200 and 350 V with a current density of 0.2–0.5 A/cm² [9]. When processing large-size workpiece, the current in the EPP circuit may increase sharply (usually up to hundreds of amperes) due to the large processing area, which will lead to a large amount of heat release. Consequently, the electrolyte temperature rises rapidly and gets boiling. The high current and electrolyte boiling will cause violent liquid splashing, which is dangerous and will deteriorate the polishing effect. In this paper, a spray electrolyte plasma polishing (SEPP) method for large-size and complex surfaces is proposed. The SEPP experimental equipment is shown in Fig. 1. In this SEPP system, a spray head is used as the cathode to replace the electrolyte bath in traditional EPP equipment. The spray head is connected to the end of a six-joint robot. During the SEPP process, the outlet of the spray head is controlled to face the workpiece surface directly. When an appropriate voltage is applied, a strong and relatively uniform electric field will be generated on the metal surface facing the cathode, which ensures the polishing effect of this area. On the surfaces far from the cathode, although there is electrolyte flowing along them, these surfaces will not be polished due to the weak electric field. In this way, only a small local area of the workpiece is being polished at a time. By SEPP method, high localization of the machined area can be achieved, and the current in the SEPP circuit is limited.
within a small range (lower than 50 Å). In the SEPP process, by accurately controlling the motion trajectory of the robot and the turntable, the polishing of the external surface of large-size and complex workpiece will be achieved.

Figure 2 shows the schematic diagram of SEPP mechanism. As shown in Fig. 2a, the workpiece is connected to the positive pole of the DC power supply, as the anode and the spray head are connected to the negative pole as the cathode. The outlet of the cathode is controlled parallel to the anode surface to be polished. The electrolyte that heated to a certain temperature (70–80 °C) is sprayed on the anode surface with a certain flow rate. Then a voltage between 200 and 350 V is applied. When the electrolyte contacts the anode workpiece, an instantaneous short circuit occurs, and the temperature of the anode rises rapidly, leading to the instantaneous electrolysis and evaporation of the electrolyte, as shown in Figs. 2c and 3b. Meanwhile, an insulating VGE consisting of bubbles is formed between the workpiece surface and the electrolyte.

The main cause of surface material removal in the SEPP process mainly includes two mechanisms. The fluctuation of the VGE makes the electrolyte constantly contact and separate from the workpiece surface [22]. First, the electrochemical reaction occurs when the electrolyte contacts with the anode surface. As shown in Fig. 2d, e, the active ions in the electrolyte electrochemically react with the metal atoms of the anode surface. Generally, electrochemical reactions including metal oxidation and oxygen evolution are as follows [11]:

\[
M - ne^- \rightarrow M^{n+} \quad (1)
\]

\[
4OH^- - 4e \rightarrow 2H_2O + O_2 \uparrow \quad (2)
\]

\[
M^{n+} + OH^- \rightarrow \frac{M_a}{M_c}(OH)_d \downarrow + H^+ \quad (3)
\]

where M is the metal element.

The second mechanism is the plasma discharge that occurs when the electrolyte and anode are separated by the VGE. The polishing system can be equivalent to the circuit, as shown in Fig. 4, where R1 represents the resistance of the electrolyte between the cathode and the anode, and R2 is the...
resistance of the fluctuating VGE. Since the VGE is almost insulated while the electrolyte is conductive, the resistance value of $R_2$ is much greater than that of $R_1$. Therefore, the dominant voltage drops are concentrated on the VGE. Due to high voltage, the electric field strength in the VGE is as high as $10^6–10^8$ V/m [23]. Electrons near the VGE-electrolyte interface are accelerated by the electric field and move to the anode surface. These electrons will strike other molecules to generate new electrons and ions, leading to an avalanche increase of electrons. Gradually, conductive channels that connect the electrolyte and the anode surface are formed, which is characterized by plasma discharge. Due to stronger local electric field, more discharge channels will be formed at the protrusions of the metal surface. As shown in Fig. 2f, electrons with high speed strike on the anode surface, causing the rapid rise of metal surface temperature and the melting of reaction products. Subsequently, the shock wave generated by plasma discharge will remove the melted portion of the surface protrusion, which exposes a new surface to continue the electrochemical reactions, as shown in Fig. 2g. Finally, a smooth workpiece surface is achieved.

During SEPP process, the decreasing rate of surface roughness mainly includes three aspects: the material removal rate, the prior material removal at the protrusions and the crater size caused by plasma discharge. Under certain polishing conditions, it can be considered that the material removal rate and the crater size are unchanged [24]. Thus, the surface roughness decreasing rate only depends on the prior material removal at the protrusions of the surface. Especially, the existence of plasma discharge craters indicates that there is a minimum achievable surface roughness theoretically. When the surface roughness is decreased to the minimum value, it will keep unchanged, that is, the decreasing rate of the surface roughness is 0. Accordingly, the differential equation between the workpiece surface roughness and processing time is established:

$$\frac{dR_a}{dt} + k(R_a - R_{a\min}) = 0$$ (4)

where $R_a$ is the surface roughness at time $t$, $R_{a\min}$ is the theoretical minimum surface roughness.

The relationship between the processing time $t$ and the spray head feed velocity $v$ is:

$$t = \frac{d}{v}$$ (5)

where $d$ is the inner diameter of the spray head, $d = 20$ mm.

Therefore, the differential equation between surface roughness $R_a$ and spray head feed velocity $v$ is:

$$\frac{dR_a}{dv} + k(R_a - R_{a\min}) = 0$$ (6)

$$\lim_{v \to +\infty} R_a = R_{a0}$$ (7)

where $R_{a0}$ is the initial surface roughness of the workpiece.

The general solution of Eq. (7) is:

$$R_a = R_{a\min} + Ce^{-\frac{v}{\tau}}$$ (8)

According to Eq. (8):

$$C = R_{a0} - R_{a\min}$$ (9)

Let $R_{a1} = R_{a0} - R_{a\min}$, the special solution of Eq. (7) is:

$$R_a = R_{a\min} + R_{a1}e^{-\frac{v}{\tau}}$$ (10)

### 2.2 Experimental system and specimen

For the convenience of experiments and subsequent surface properties' detection, several GH3536 superalloy plates, 200 mm × 100 mm × 5 mm in size, manufactured by SLM were used as the specimens. Prior to experiments, the initial surface roughness of the specimens was kept as consistent as possible ($13.5 \mu m \leq R_a \leq 14.0 \mu m$).

Ammonium sulfate electrolyte with a mass fraction of 3% and a pH of 5 was used in the experiments. In addition, some complexing additives suitable for electrolytic plasma polishing of GH3536 superalloys were added to increase the glossiness of the specimens.

| $v$ (mm/s) | 0.075 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 |
|-----------|-------|-----|-----|-----|-----|-----|-----|-----|-----|
| $R_a$(μm) | 0.174 | 0.228 | 0.565 | 1.191 | 1.863 | 2.959 | 3.743 | 4.519 | 5.544 |
In order to accurately measure the surface roughness specimens, a roughness tester of the type MarSurf M300C was used. And a confocal laser scanning microscope (CLSM) of type Smartproof 5 was used to evaluate the three-dimensional surface morphology of the specimens. Moreover, microscopic surface morphology observation and chemical element analysis were performed through the scanning electron microscope (SEM) of the type JSM-IT500LA equipped with energy dispersive spectroscopy (EDS).

3 Results and discussion

3.1 Regularity of surface roughness with cathode feed velocity

Preliminary investigation was performed to assess the influence of the velocity of the spray head upon the surface roughness. The distance between anode and cathode was 30 mm, and the applied voltage was 320 V. The temperature and flow rate of the electrolyte were controlled at 70 °C and 15 L/min, respectively. Table 1 shows the surface roughness of the polished specimens at different spray head feed velocity. According to Formula (10), OriginPro software is used for nonlinear fitting analysis, and the dependence of the surface roughness \( R_a \) upon the spray head feed velocity \( v \) is shown in Fig. 5a. The experimental data points are distributed near the curve and the adjusted \( R^2 \)-squared value is 0.99611, which means that the curve fitting effect is satisfactory.

The result in Fig. 5a shows that lower velocity achieves better surface roughness. This is because there is more time to remove materials from the metal surface with a lower velocity. Different spray head feed velocities correspond to different treatment time; the dependence of surface roughness upon the treatment time is shown in Fig. 5b based on Formula (5). According to the above polishing mechanism, the SEPP process starts by removing materials at the protrusions of the specimen surface. As the surface is gradually flattened, the thickness of the removed layer decreases when removing the same amount of materials, i.e., \( h_1 > h_2 \) shown in Fig. 2e, g. Therefore, the surface roughness decreases rapidly at the beginning of the SEPP process and then continuously decreases to a minimum value.

According to the above analysis, a smaller spray head feed velocity means a longer treatment time. Therefore, in practical application, it is necessary to comprehensively consider the relationship between the desired surface roughness and polishing efficiency to choose a suitable processing speed. For specimens in this experiment, the suitable spray head feed velocity is between 0.1 mm/s and 0.2 mm/s.

3.2 Influence of the process parameters

During the SEPP process, several parameters may significantly affect the final surface roughness of the specimens. A series of single factor experiments were designed to study the influences of parameters on the surface roughness, such as the distance \( L \) between the anode and the cathode, the voltage \( U \), the temperature \( T \) and flow rate \( Q \) of
the electrolyte. The experimental items and parameters are shown in Table 2.

The applied distance $L$ between anode and cathode is changed from 20 to 50 mm (with an interval of 5 mm). Figure 6a shows the surface roughness $R_a$ under different distance $L$. In the range of 25–50 mm, the surface roughness decreases with decreasing distance $L$. It can be explained by the increase of the electric field and the current in the SEPP circuit (Fig. 4). Higher current is an indicator that more materials are removed, and thus a lower surface roughness is achieved. When the distance $L$ is reduced to 20 mm, the VGE will fluctuate violently due to the further enhancement of the electric field, which may be confirmed by the frequent strong sparks observed in experiment, as shown in Fig. 7b. Therefore, the polishing process becomes unstable, resulting in a higher surface roughness than the result when the cathode–anode distance is 25 mm.

As shown in Fig. 6b, when the applied voltage is lower than 300 V, the surface roughness decreases with increasing applied voltage. The rise of the applied voltage will increase the energy of the particles reaching the anode surface. Thus, the material removal rate improved, and a lower surface roughness was obtained. However, the higher energy also means a large amount of heat release, which will lead to VGE thickness and decrease the current. Moreover, a thicker gaseous envelope may hinder the electrochemical reaction between the electrolyte and the anode surface. Therefore, the final surface roughness is increased when the applied voltage exceeds 340 V. Lower surface roughness was obtained when the applied voltage $U$ was 300 V and 320 V.

It can be seen from Fig. 6c that when the electrolyte temperature $T$ is between 70 and 80 °C, a lower surface roughness is achieved. A lower electrolyte temperature requires more energy to maintain the SEPP process. Because of the constantly moving

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**Fig. 6** Surface roughness $R_a$ dependence upon 
(a) the distance $L$ between anode and cathode, 
(b) the applied voltage $U$, 
(c) the electrolyte temperature $T$, 
(d) the electrolyte flow rate $Q$

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**Fig. 7** Experimental phenomena: 
(a) normal SEPP process, 
(b) strong spark was observed
spray head, the processing time for each area of the specimen is kept unchanged. The treated area will not be polished sufficiently in a lower temperature environment. Moreover, decreasing the temperature will reduce the activity of the ions associated with electrochemical reactions in the electrolyte. Therefore, the final surface roughness value of the specimen is significantly increased when the electrolyte temperature is lower than 65 °C. As the temperature increases from 80 to 85 °C, the electrolyte appears locally boiling; the VGE gets thicker; and some electrolyte components will quickly decompose and volatilize, which will decrease the material removal rate and increase the obtained surface roughness.

As shown in Fig. 6d, the surface roughness presents a decreasing trend with the flow rate $Q$ increasing from 5 to 12.5 L/min. When the flow rate is between 5 and 7.5 L/min, the electrolyte exhibits a distinct parabolic curve. The treated area is below the spray head, where the electric field is lower than that of the area with the same height as the spray head, leading to a higher surface roughness. As the electrolyte flow rate exceeds 15 L/min, excessive flow rate will lead to high pressure of the electrolyte reaching the anode surface. Based on the analysis above, VGE is composed of numerous small bubbles and is fluctuating, which can be easily affected by external pressure, making the polishing process unstable and discontinuous. Ablyaz et al. [25] reported that a lasting VGE cannot be formed under high electrolyte pressure. Therefore, a higher surface roughness is obtained compared with the results when the electrolyte flow rate is between 10 and 12.5 L/min.

### 3.3 Surface properties

To gain further insight into the polishing effect of SEPP technology, another experiment was conducted with more suitable parameters, as shown in Table 3, based on the results of the above experiments. Surface properties such as surface roughness, micro morphology, chemical composition, and surface hardness of the GH3536 specimen before and after polishing were detected and analyzed.

Figure 8a shows the surface roughness $R_a$ and $R_z$ of the pre-polishing (PP) specimen and the SEPP-treated specimen. As shown in Fig. 8a, the SEPP process can significantly decrease the surface roughness from $R_a = 13.93 \, \mu m$ to 0.107 \, \mu m, and the surface roughness $R_z$ is reduced from 78.18 \, \mu m to 1.130 \, \mu m. Figure 8b, c show the micro profiles of the PP and the SEPP surfaces. The CLSM images of the PP and the SEPP surfaces

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**Table 3** The experiment parameters

| $V$ (mm/s) | $L$ (mm) | $U$ (V) | $T$ (°C) | $Q$ (L/min) |
|------------|----------|---------|----------|-------------|
| 0.1        | 25       | 320     | 75       | 12.5        |

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Fig. 8 Comparison between the PP surface and the SEPP surface: a the surface roughness; b, c the micro profiles; d, e the CLSM images; f the surface appearance
are presented in Fig. 8d, e, respectively. The surface appearances of the PP and SEPP specimens are presented in Fig. 8f. Apparently, the results in Fig. 8 show that a smooth and shiny surface can be achieved by the SEPP technology.

As shown in Fig. 9a, there is a large amount of spherical powders on the PP surface of the specimen, which is a typical defect of additive manufacturing parts. Comparing the SEM results in Fig. 9a, b, it is significant that both the protrusions and the spherical powders are removed, and there are no obvious machining traces on the SEPP surface. In the SEM image with 5000× magnification, dense plasma discharge craters can be observed, which is a confirmation of the existence of plasma discharge.

Comparing the results in Fig. 10a, b, after SEPP treatment, the O element on the specimen surface disappeared, and the content of Al element is decreased. It can be explained by the fact that the oxides (e.g., Al₂O₃) on the metal surface were removed by SEPP process. Further analysis of the EDS spectrums shows that the contents of Ni, Cr, and Fe elements are increased because the oxides and dirt on the surface were removed, and more Ni, Cr, Fe elements were exposed on the metal surface. Moreover, the Cr and Fe elements are more active than Ni element. The ions in the electrolyte will preferentially react with elements with higher activity, and then the reaction products are removed by plasma discharge, resulting in the detected Ni content to have increased more than Fe and Cr elements.

Figure 11 corresponds to the Rockwell hardness of the PP surface and the SEPP surface. Select 9 points on the PP surface and SEPP surface for hardness testing, respectively. The test force of Rockwell hardness tester was set as 150 kgf. It is obvious that the Rockwell hardness values of the initial surface of the specimen are lower and show poor consistency. After the SEPP process, the Rockwell hardness values of the specimen is approximately 30 HRC and show good consistency due to the smooth and flat surface.
face roughness can be achieved when the feed velocity is between 0.1 and 0.2 mm/s. Further slowing down the feed velocity will continually decrease the surface roughness. A significant reduction of the surface roughness can be achieved when the parameters are $L = 25$ mm, $U = 300–320$ V, $T = 70–80$ °C, and $Q = 10–12.5$ L/min. Especially, considering the coupling between factors, an orthogonal experiment is planned in the following study to determine the primary and secondary factors affecting the final surface roughness, and to obtain the optimal parameter combination ultimately.

3. In addition, SEPP technology was found to decrease the surface roughness from $R_a = 13.93$ μm to 0.107 μm, and the treated specimen was smooth and bright without obvious machining traces. The oxides and dirt on the surface were removed and the contents of Ni, Cr, and Fe were increased. The surface hardness of the polished specimen increased and showed good consistency, compared with the initial specimen.

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