Surface treatment of Ti6Al4V parts made by powder bed fusion additive manufacturing processes using electropolishing

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Abstract

This paper investigated the use of electropolishing on the surface treatment of the Ti6Al4V parts made by the powder bed fusion processes including direct metal laser sintering (DMLS) and electron beam melting (EBM). A non-aqueous alcohol based electrolyte was used, and the relationship between the process and surface roughness was evaluated. Based on the results, the feasibility of electropolishing as a potential alternative post-surface treatment for additive manufactured metal parts was discussed.

Introduction

Surface quality has long been a haunting issue for powder bed fusion additive manufacturing (PBF-AM) processes. In addition to the staircase effect which is intrinsic to most additive manufacturing (AM) processes, PBF-AM processes also faces additional challenge. As the powder in fabrication area is melted, heat transfer occurs inevitably, which causes the powder adjacent to the fabricated area to be partially sintered and attached to the part surface. The surface sintering effect not only reduces the geometrical accuracy of the fabricated geometries, but also creates surface defects which could serve as crack initiation sites. For structures with large surface-to-volume ratios such as cellular structure or honeycomb structures, surface properties could sometimes dominate the overall performance of the structures, which further highlights the significance of the issue. On the other hand, fatigue performance is of critical importance to many applications in aerospace, automobile and bi-devices, which are widely regarded as the most promising targeting markets of AM technologies. Therefore, the surface quality improvement of PBF-AM fabricated structures is of critical importance for the future adoption of the technologies.

Various efforts have been reported in the attempt to improve the surface qualities of PBF-AM parts. It has long been recognized that the surface quality of the PBF-AM parts are largely influenced by process parameters [1-3]. In some works, active process control such as surface re-melting [2, 4, 5], selection of fine powder [6] and the use of optimized parameters [7, 8] have been utilized to improve the surface quality of the parts. However, due to the intrinsic characteristics of the process, these in-process surface quality control methods could only achieve limited effects.

On the other hand, multiple post-process surface treatment methods have also been used to improve the geometrical accuracy of the final parts. Traditional surface treatment methods such as machining [9, 10], mechanical polishing [11], abrasive flow polishing [12], chemical milling [2, 13] and electroplating [11] have been investigated for their efficiency in treating AM fabricated parts, and they achieved mixed results. With most of the surface treatment processes, geometrical complexity of the treated parts is the major challenge. For example, mechanical polishing and machining could be difficult to apply on undercut features and internal features, and abrasive flow polishing could usually only achieve desired surface finish on selected surfaces due to the directional flow of the abrasive fluid. Chemical milling and electroplating both rely on the mass transportation between a polishing fluid and the workpart, therefore are both capable of accessing all surfaces as long as they are external. Chemical milling is capable of removing surface materials at a wide range of controlled rate, and it also leaves minimum residuals on the treated surface as long as the stirring or mass transportation is sufficient. However, chemical milling often does not completely eliminate surface features due to the isotropic etching effect. As shown in Fig.1, after chemical milling, the surface bumps and extrudes of the thin lattice Ti6Al4V part made by electron beam melting (EBM) was reduced to small sharp-tip features instead of disappearing completely [13]. As a result, the fatigue performance of such structures are only marginally improved and still considerably lower than that of the equivalent material with machined surfaces. On the other hand, the electroplating process is often used to alter the surface properties of a part by coating a thin layer of material on the part surface. Similar to anodizing process, electroplating process is based on both Faraday's Law and the Laws of mass transportation. With Faraday's Law, the rate of electroplating reaction is influenced by the distance between the electrodes. Therefore, when setup properly, the extruded features on a surface is expected to have higher electroplating rate compared to the other part of the surface, which could potentially leads to higher ion deposition rate. As a result, electroplating could actually increase the severity of the surface roughness. To this end the electroplating process is not suitable for the improvement of surface finish.

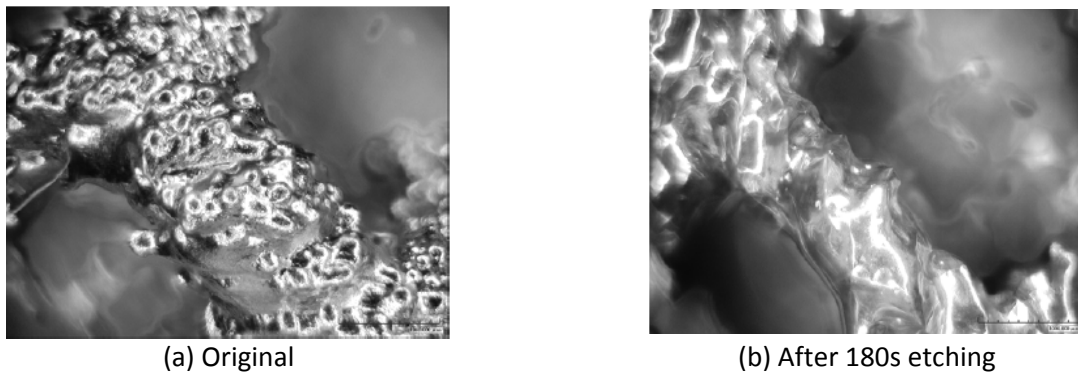| (a) Original | (b) After 180s etching |

Fig.1 Chemical milling effect on Ti6Al4V thin feature [13]

Electropolishing is in principle the reverse of electroplating. The treated part is not connected as a cathode but as an anode, and when voltage is applied, the anode will be polished by the removal of surface metal particles into electrolyte as shown in Fig.2. Driven by electrical potential, the ionized particles from the treated workpart will move towards the cathode enabled by the pathway provided by

electrolyte. Since the process is the reverse of electroplating, electropolishing is also driven by Faraday's Law, which means that the extruded features on the surface will be selectively polished more severely. Therefore in principle electropolishing could effectively achieve selective smoothing of surface features and the improvement of surface finish. This features, combined with the ability to access complex surfaces, making electropolishing a desirable candidate for the surface treatment of AM parts.
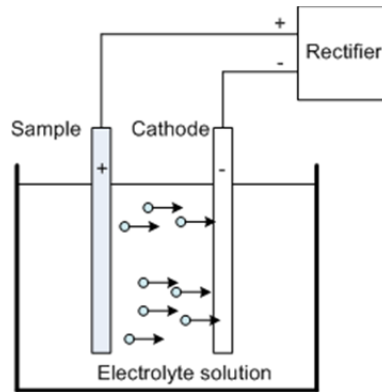

Fig.2 Electropolishing process

To date there has not been any literature that reported the use of electropolishing on AM surface treatment, therefore, in this work preliminary experiments were performed to evaluate the feasibility of this concept.

Understanding Electropolishing

Even though the basic principle of electropolishing is Faraday's Law, in practice the mass transportation is also a critical issue. As the ion particles forms from the workpiece, they will need to be carried away sufficiently by the flow of the electrolyte. Also, in some electropolishing processes, a compact oxide layer could form at the newly polished surface [14, 15]. If the compact film could be readily dissolved by the electrolyte chemicals with the assist of electrical field, then the electropolishing process could continue with a characteristic equilibrium oxide layer on the surface of the anode that is sufficiently thin to allow for cations to diffuse. The thin oxide layer also prevents the etching effect when acidic solution is used since no selective chemical reaction at grain boundaries would take place. The higher viscosity and dissolution concentration at the valleys of the anode surface would eventually contribute to the smoothing effect of the electropolishing. There exist a threshold voltage potential for the breakdown of the oxide layer, which would result in significant gas generation and is usually avoided in the electropolishing process due to its tendency to deteriorate the polishing quality [16]. Therefore, usually the electropolishing process takes place at a mildly high voltage in order to sustain sufficient anode ionization while avoiding the onset of decomposition reaction of the oxide layer. Beside oxides, reaction products from the electropolishing process could also deposit on the workpart surface under the effect of diffusion, which is also undesired since it creates a barrier for mass transportation between the workpart and the electrolyte. A common method used to overcome the challenge of excessive oxide and

precipitation layer formation on the workpart surface is the stirring or agitation of electrolytes [15, 17-18].

For most electropolishing processes, there exists a particular potential-current relationship that determines the optimum electropolishing parameters. This relationship is largely determined by the type of material and electrolyte used in the process. In a typical relationship, the current would increase approximately linearly at low potential levels until it reaches a threshold, after that there exists a plateau stage in which the current keeps more or less constant as the potential increases. At this stage, the electropolishing is dominated by mass transportation phenomenon. The ions diffuse through the surface layer at a stable rate, creating electropolishing current that is little dependent on the voltage level. This is widely regarded as the suitable envelop for electropolishing [19, 20]. When the potential is sufficiently high, the current will start to increase again in a more drastic rate, which is referred to as chemical pitting [17, 18]. At this stage significant breakdown of either the electrolyte or oxide layer occurs, and the resulting part surface is often affected by pitting effect. In short, the first step of the electropolishing experiment would be to establish the potential-current relationship for the chosen electrolyte and identify the plateau stage.

It was also reported that other factors such as temperature [14, 21] and water content in the electrolyte [17] could have significant effect on the electropolishing effects. Higher temperature could promote the mass transportation and therefore facilitate the polishing reaction, however it could also cause unwanted decomposition reactions, therefore has a mixed effect on electropolishing. On the other hand, water is considered to be detrimental to electropolishing since it could result in excessive oxidation of the surface and the formation of hydrogen gas, which are both unwanted for the process.

Recently, more sophisticated electropolishing by pulse AC current has been reported [22, 23]. It was suggested that via the switching of electrode polarity, the process could replenish the ions in the electrolyte more efficiently and help the process to maintain high limit current. Due to the lack of high accuracy AC waveform rectifier, this alternative was not investigated in the current study.


Experiments and Discussions

In this study, effort was focused on the investigation of electropolishing with titanium Ti6Al4V parts made by electron beam melting (EBM) process. Titanium alloys have been notoriously difficult to process with chemical and electrochemical methods. Due to the existence of the highly stable oxide thin film on the surface, traditionally only a few acids could effectively react with titanium alloys, including hydrofluoric acid and perchloric acid [24, 25]. These chemicals are highly hazardous and could cause serious accidents if not used with extreme care. Recently, a non-toxic and non-explosive recipe has been patented for the effective electropolishing of titanium alloys [21]. This electrolyte includes ethanol, isopropyl alcohol, aluminum chloride and zinc chloride. This non aqueous electrolyte solution was successfully used to polishing both CP-Ti and Ti6Al4V alloy and achieved surface finish of $R_a < 100 \mu m$. This electrolyte solution could be easily realized in regular lab environment with minimum safety concerns, and was adopted for further investigation in this study.

Ti6Al4V is the most widely used material for EBM process, which sees many potential applications in aerospace and biomedical industries. The surface finish of typical EBM parts ranges between 20-30μm. The surface finish of the top surface is usually slightly better, while the sides are usually rougher. Due to the use of relatively coarse powder compared to the laser based PBF-AM process and the larger size of the electron beam, the surface quality issue is more significant with the EBM process. In the current study, sample plates with flat surface area of 5mmx20mm was designed and fabricated by the EBM S400 system using the default Ti6Al4V melt theme. The sample was oriented in the build so that the large flat surfaces are oriented vertically. Fig.3 shows one of the sample part as well as the surface profile measured by a Zygo surface profilometer. The surface finish of the large flat surface was about R_a=23μm, which agreed with the literature.
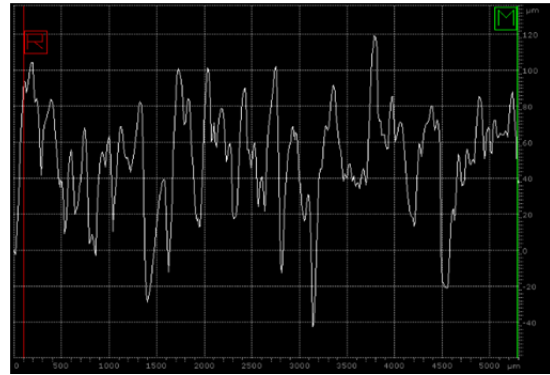
(a) Sample part   (b) Surface profile
Fig.3 EBM sample for electropolishing experiment

The test setup was designed carefully to achieve accurate control of the electrode distance, dipping depth and agitation of the electrolyte. As shown in Fig.4, a glass beaker was used as container of electrolyte, and the electrodes were fixed in a holding plate, which was in turn fixed by a central threaded rod and three guide bars. Through the adjustment at the central threaded rod, the holding plate could move up or down by sliding along the guide bar, therefore achieving accurate control of the dipping depth of the electrodes. The guide bar also served as electrical path for easy clamp of crocodile clips. A magnetic stirring bar was placed at the bottom of the beaker to provide agitation during the electropolishing. For each experiment, roughly 300mL of electrolyte was used. The electrolyte was made by following the recipe provided in previous literatures: 1L of electrolyte should consist of: 700mL of ethanol, 300mL of isopropyl alcohol, 60g $AlCl_3$ and 250g $ZnCl_2$. A DC power supply with maximum potential of 75V was used for all experiments. During the experiment, the beaker was placed in ice water bath in order to maintain low temperature.

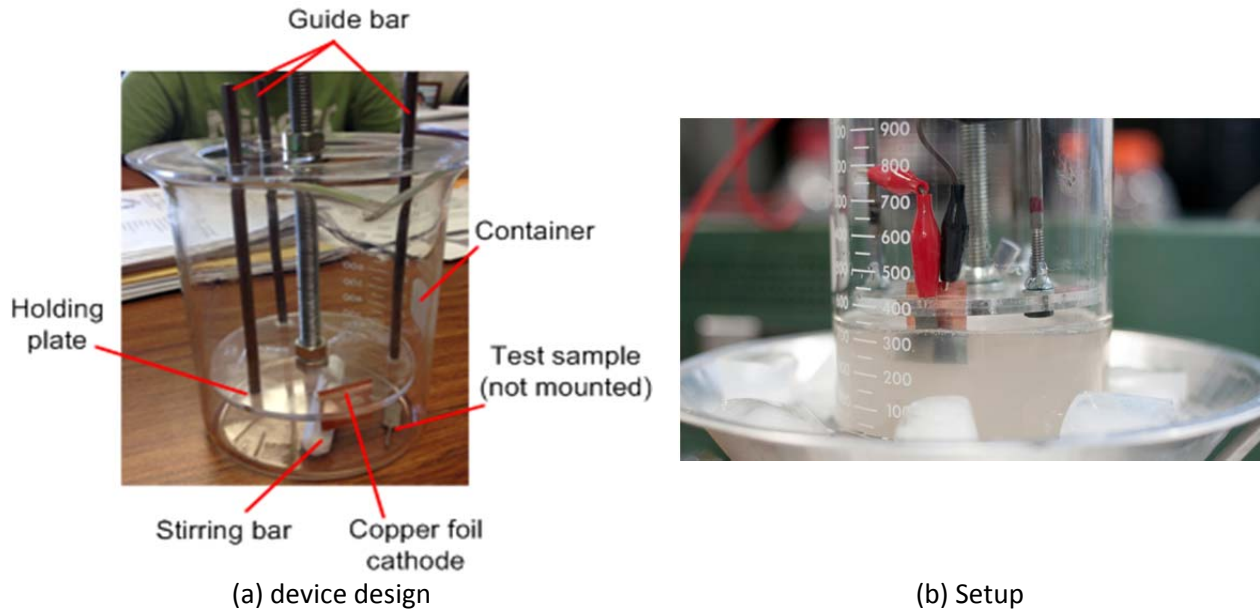(a) device design   (b) Setup

Fig.4 Electropolishing experimental setup

Three electrode distances were investigated for the study, which were 5mm, 7.5mm and 10mm. The potential-current curve was obtained by gradually increase the potential values at each electrode distance setup and record the change of currents. A constant stirring setting was used throughout all the tests, although the control was not accurate and therefore no accurate rpm was recorded. Fig.5 shows the relationship for all three setups. None of the curve exhibited significant plateau, however, there existed a rather drastic slope at around 65-70VDC for all three setups, and the increase of current before this threshold was rather mild. It was also observed that accompanied the sharp rise of current was the formation of gas phase. Therefore, it was speculated that 70VDC was the threshold of significant chemical pitting, and while no apparent plateau was present, the electropolishing would likely to be both stable and rapid at around 50-60VDV range. With that in mind, an electropolishing potential of 55VDC was selected for the subsequent experiments.

(a) 5mm   (b) 7.5mm   (c) 10mm
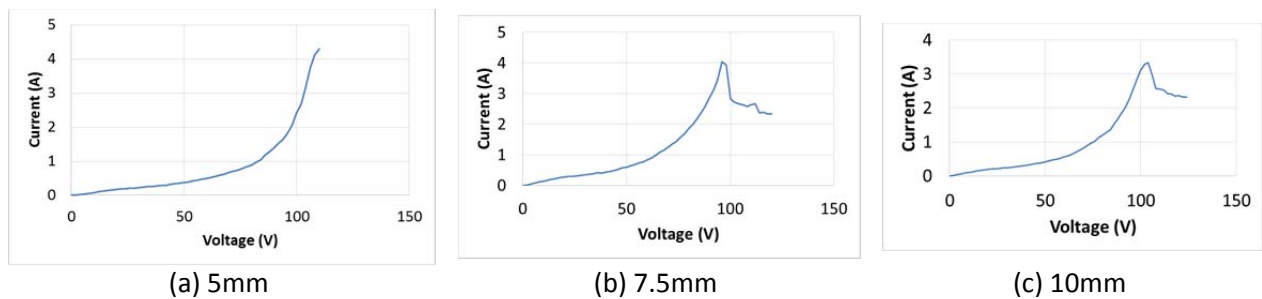
Fig.5 Potential-current curve at different electrode distances

Using the three electrode distance setups, electropolishing experiments were carried out with different total polishing time. It was found in the preliminary trial that considerable reaction production accumulation as well as heating occurred during the experiments. Therefore, in order to alleviate this

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issue, an artificial electropolishing waveform was introduced. The electropolishing would be carried out for 60 seconds, followed by a 10 seconds break with no potential, so the agitation could have more sufficient time to re-balance the electrolyte and wash away the surface contaminants. It was also worth noting that excessive agitation also seemed to affect the process by creating significant turbulence and vortex in the flow. An stirring setting of about 400rpm was set, although it was not strictly kept constant during some of the experiments in order to more effectively remove the surface contaminations.

The relationship between the final surface finish and the electropolishing setup is shown in Fig.6. No apparent improvement could be observed for short electropolishing time. However, significant difference occurred when polishing time was 20 minutes. As expected, the setup with 5mm electrode distance resulted in the most significant improvement of surface quality likely due to the larger current and faster reaction rate. On the other hand, the polishing rate of setup with 10mm electrode distance did not appear to have significant effect on the surface even after elongated polishing time. It is worth noting that significant non-uniformity was also observed for most of the samples. Within the same sample, there exist areas that had better surface finish than the others, which could be readily explained by the observed turbulence of the electrolyte flow. From Fig.4 it is easy to realize that the setup does not create a linear laminar flow bur rather vortex flow, therefore different areas of the flat part is subject to different shear and normal pressures from the electrolyte flow. Fig.7 shows some of the final parts, and it could be easily seen that the edges of the parts are polished more significantly, which are the areas that undergoes more pronounced flow agitations. The best surface finish achieved by the 5mm+20minute sample was about R_a=6μm, which was a significant improvement compared to the original sample.
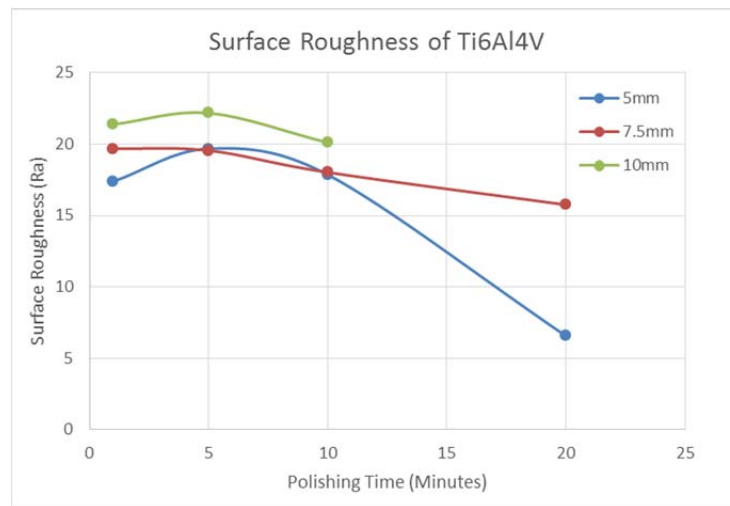

Fig.6 Effect of electropolishing parameter on the surface quality

(a) 5mm+10 minutes   (b) 7.5mm+10 minutes   (c) 5mm+ 20 munites

Fig.7 Samples after electropolishing

The experiment also revealed some issues. From Fig.7(c), it could be clearly seen that a rather serious shape accuracy loss occurred on the processed parts. This could be contributed by both the non-uniform electrolyte agitation and the insufficient control of the total amount of electropolishing. Another issue was the repeatability of the experiments. Without accurate control of stirring speed and temperature, the electropolishing could still exhibit significant difference even if the other parameters are well controlled. Fig.8 shows a sample that after electropolishing at 5mm+20 minutes setup, and the surface finish at some area was R_a<100nm. Further study is under way to redesign the experimental setup and to achieve better control of the process in order to duplicate the good results and identify critical parameters.

Fig.8 Electropolished sample with extraordinary surface finish

Conclusion

The preliminary study has shown that electropolishing possess potential to be used as an effective surface treatment method for PBF-AM fabricated parts. Ti6Al4V was chosen as the material in this study because of its popularity with PBF-AM processes as well as its difficult with traditional processes, and the results showed significant promise. Further studies are ongoing that need to achieve better control of the electropolishing process and better understanding of the key parameters.

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