Further study of the electropolishing of Ti6Al4V parts made via electron beam melting

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Abstract

In this study, the effect of various parameters including the voltage, current, polishing time, temperature and electrode spacing on the electropolishing quality of Ti6Al4V samples made via electron beam melting was investigated using specialty designed research setups. The relationships between these process parameters and the surface roughness of the parts were established experimentally.

Introduction

In the previous study, the authors investigated the use of non-aqueous electropolishing as a potential method for the surface treatment of Ti6Al4V parts fabricated via electron beam melting (EBM) [1]. A surface finish of 6µm was achieved via experiments, although the uniformity of the surface polishing was rather unsatisfactory. Furthermore, through the investigation of relationships between several process control variables such as electrode spacing, polishing time and the surface finish, it was concluded that this method could potentially achieve efficient surface finish improvement for EBM Ti6Al4V parts and structures [1].

Various literatures have reported the high sensitivity of the electropolishing process on various process parameters such as temperature, agitation and electrical excitation energy [2-5], which is largely due to the underlying polishing mechanisms. For a surface feature that possesses both macroscopic waviness (>1µm) and microscopic roughness (<1µm), which are driven by different mechanisms [3]. While macro-smoothing are primarily dominated by the electrical current level, the micro-smoothing is dominated by mass transportation of dissolved anions (i.e. diffusion). For most electropolishing practices, both macro- and micro-smoothing are desired, as macro-smoothing removes large surface roughness, and micro-smoothing result in brightening of the surfaces. Therefore, as diffusion is strongly influenced by both temperature and the fluid flow that carries the reaction species away from the electrodes, the electropolishing effects are affected consequently.

Multiple works have investigated the electropolishing of titanium and titanium alloys using various electrolyte recipes such as methanol-sulfuric acid [4], methanol-buthanol-perchloric acid [6], hydrofluoric acid-lactic acid-sulfuric acid [7], perchloric acid-acetic acid [8, 9], perchloric acid-ethylene glycol monobutylether-methanol [10], sulfuric acid-hydrofluoric acid-acetic acid, sulfuric-phosphoric acid-hypophosphite-tartrate, anhydrous sulfamic acid-formamide and sulfuric acid-KF-ammonium bifluoride [11]. During the electropolishing process, the polishing reaction and oxidation reaction as shown in Eq.(1) often take place simultaneously. While the

polishing reaction dissolves titanium atoms from anode parts, the oxidation (passivation) reaction forms a thin and uniform TiO₂ layer on the part surface that limits the titanium anion diffusion into the electrolyte solution. As shown in Fig.1, for titanium the voltage-current electropolishing characteristic often exhibits an initial slope followed by a plateau stage in which the electropolishing current remains largely constant. It could be expected that the dominant polishing mechanism is macro-smoothing during the initial stage, which changes into a combined macro-/micro-smoothing mechanism that is desired for the process. On the other hand, as shown in Fig.1, there exist a complex balance between various reactions during the plateau stage, including the diffusion of the dissolved titanium anion into the electrolyte through the passive oxide surface film under electrical potential, the build-up of the titanium oxide layer and the breakdown of this oxide layer. Due to the high propensity of passivation, titanium alloys often requires highly corrosive acids such as hydrofluoric acid and perchloric acid in order to effectively break down the oxide layer during the polishing process, which pose serious health and safety hazards.



Fig.1 Typical electropolishing characteristic of titanium

The use of non-aqueous electrolyte was suggested to provide a possible solution, as the existence of water strongly promotes the formation of stable titanium oxide layer [4]. Several anhydrous titanium electrolyte recipes have been developed, including the previously mentioned sulfamic acid-formamide [11] and ethanol-isopropyl alcohol-zinc chloride-aluminum chloride [12]. The latter was of specific interest to the authors as all the ingredients are commonly available and pose less overall health and safety hazards. It was reported in the previous study that this electrolyte does not eliminate the formation of oxide layers. Instead, a viscous surface layer consists of both titanium oxide and titanium salt was formed, which can be relatively easily removed via electrolyte agitation [12].

In this study, an improved electropolishing device was designed and built, which allows for improved control with the temperature and the electrolyte agitation. Utilizing the improved device, experiments were performed to characterize the electropolishing process and to optimize the polishing parameters.

Experimental setup

The improved experimental setup is shown in Fig.2. The main reaction chamber was a custom built 70mmx85mmx85mm glass tank with two fluid inlet/outlet, which was fixed on a wooden substrate during the experiments. The electrodes were connected to an Instek PSW 160-14.4 multi-range DC power supply. The electrolyte agitation is driven by a Masterflex 7523-40 L/S peristaltic pump and circulates through a custom built Liebig condenser as the heat exchanger for heating/cooling of the electrolyte. The temperature of the water used for heating/cooling was controlled by a water tank with automatic heating/cooling control (not shown in Fig.2). The heating/cooling was designed to be indirect as it was found to be difficult to find an economical direct cooling solution with the high-purity ingredients used for the study. In addition, a thermocouple was placed into the electrolyte solution with glass protection in order to monitor the temperature variation during the electropolishing process.



Fig.2 Electropolishing experimental setup

During the experiments, the peristaltic pump was set at a constant flow rate of approximately 1.3L/min, which was considered sufficient as the electrolyte solution volume used for each experiment was approximately 400mL. As the heated water tank possesses a rather large heat sink, the temperature setup was done prior to the experiments to ensure sufficient heating of the electrolytes. However, it was found during preliminary experiment that there still exist considerable temperature drift in the electrolyte solution even with the use of the cooling mechanism shown in Fig.2, which was largely due to the large voltage and current used in electropolishing. Therefore, the electropolishing process was set to pause for 60s for each 60s of polishing to allow for additional time for cooling, which was shown to be able to limit the temperature drift to within 10°C for all the experiments performed. The control of the on/off of the power supply was realized via a LabView program.

For the electropolishing, Ti6Al4V bars with 20mmx8mmx2mm size were fabricated in Arcam S400 system using standard Ti6Al4V solid theme. All the samples were fabricated in the same orientation as shown in Fig.3a. A grip end was also designed for each sample to facilitate the gripping of the parts during electropolishing. A copper foil was used as the cathode, with dimensions sufficiently larger than the processed samples. After polishing, the surface finish of the samples were measured by a Dektak 8 profilometer. All the measurements were done with a 2.5mm measurement length around the central area of the polished surface as illustrated in

Fig.3b. Beside the surface finish measurement, the weights of the samples were also measured before and after the electropolishing in order to evaluate the material removal.



Experiments and Discussions

The electropolishing characteristics of the experimental setup was evaluated at multiple levels of temperatures in order to determine reasonable ranges of work envelope. Fig.4 shows the voltage-current curves of the experimental setup under three different temperatures ranging from 25°C to 53°C. It could be clearly observed that temperature has a significant effect on the electropolishing process. With only 30°C difference, the polishing speed could vary by 100-300% depending on the voltage level, which shows the importance for temperature control during this process. It was also found that very little reaction took place when the electrolyte temperature was set at 4°C, which can be readily explained by considering the fact that the Nernst potential is significantly influenced by temperature. In addition, no plateau stage was observed, which agreed with results from previous study [12]. This has two potential explanations. First, due to the lack of formation of the stable oxide surface layer, the process was not hindered significantly. Second, the reaction might had not reached the diffusion rate limit due to the sufficient electrolyte agitation. After referring to the previous results, it was determined that further electropolishing experiments would be performed at the 60-80VDC range.



Fig.4 Electropolishing characteristic curve for the experimental setup In the evaluation of effects of different process parameters on the electropolishing effects, the experiment design is shown in Table 1. For all the experiments, the electrode spacing (i.e. distance between the cathode and the anode) was kept constant at 5mm, which was identical to the setup used for process characterization. Two levels of voltage (60V, 80V), two levels of temperature (27°C, 38°C) and three levels of polishing time (5min, 10min, 20min) were investigated, and a full factorial design was performed.

Sample	Voltage (V)	Temperature (C)	Time (min)
1	60	38	20
2	60	38	10
3	60	38	5
4	80	38	20
5	80	38	10
6	80	38	5
7	60	27	20
8	60	27	10
9	60	27	5
10	80	27	20
11	80	27	10
12	80	27	5

Table 1 Electropolishing experimental design

Fig.5 shows the samples after the electropolishing. The effect of electropolishing was visually discernable. With longer polishing time, the loss of shape also becomes more significant. Temperature also appeared to be clearly more dominating compared to voltage.



Fig.5 Samples after electropolishing

Fig.6 shows the surface finish measurement results for the polished surface under each process conditions. It could be seen that when the sample was process at 80V+38°C condition, a minimum surface roughness of 2.3µmRa was achieved, although it did not appear with the 20min sample. In fact, it was observed in most samples that the polishing time did not appear to have a monotonous effect with the surface finish. This might be caused by the repeated use of the electrolyte solution for each voltage and temperature combination, as well as chemical pitting [3] in the case of the 80V+38°C group. The repeated use of solutions due to the limitation of available resources would result in gradual change of the solution composition in the electrolytes, which might potentially result in the promotion of stable oxide layer formation

at specific compositional ranges. Although preliminary study by the research group did not observe significant change with the voltage-current electropolishing characteristics between fresh and used electrolyte solutions, further studies are needed to clarify about this. Another possible cause might be attributed to the measurement process. As shown in Fig.7, the surface profile measured by the profilometer for the 80V+38°C+20min sample actually exhibit very smooth topology. However, due to the loss of geometry, the sample could not be leveled well enough during the measurement, which result in a macroscopic profile skew that could become significant over a 2.5mm measurement range.



a. As-received sample b. Polished sample Fig.7 macroscopic skew of the sample surface finish measurement

It was also interesting that the back surface of the samples were also polished. Fig.8 shows the surface roughness of the back surface of the samples. The surface finish on the back surface exhibited more consistent trends in relation to the polishing time, and the best surface finish of 0.6µmRa was obtained from the 80V+38°C+20min sample. Also, no significant pitting was observed, which suggests that the reaction on the back surface might be more controllable.



Fig.8 Surface roughness of the back surface

The measurement results of weight loss for all the samples is shown in Fig.9. The results of weight loss verified that the material removal effect is proportional to the polishing time, which suggests that chemical pitting might in fact occurred in most of the samples. The weight loss for the high voltage and high temperature group was very significant, with a maximum loss ratio of about 30%. On the other hand, it appears that the material removal rate could be accurately predicted. Therefore, it might be possible to accommodate this during the design of the samples in the future.

Fig.9 Weight loss of electropolishing

Conclusions

In this study an improved electropolishing device was implemented, which realized better control of electrolyte agitation and temperature for the electropolishing process. After implementing the new setup as well as control process pause, a 10°C temperature drift was observed during each electropolishing experiment. Through voltage-current characterization it was speculated that the electropolishing performed in this study was dominated by macrosmoothing mechanism. The electropolishing was able to achieve significant surface finish improvement from over 20 μ m with original parts down to <1 μ m, although the uniformity of the polishing is still low. Further studies are needed to optimize the process and to demonstrate the

mechanical benefits of the processed samples via fatigue testing, which is currently undergoing by the authors.

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