

# **Electropolishing of Ti6Al4V Parts Fabricated by Electron Beam Melting**

L. Yang<sup>1</sup>, Yan Wu<sup>1</sup>, Austin Lassell<sup>1</sup>, Bin Zhou<sup>2</sup>

<sup>1</sup>Department of Industrial Engineering, University of Louisville, Louisville, KY 40292

<sup>2</sup>Department of Mechanical Engineering, Tsinghua University, Beijing 100084, China

## **Abstract**

In this study Ti6Al4V samples fabricated via electron beam melting were subjected to electropolishing under various polishing conditions using the re-designed in-house electropolishing device. The surface finish of the processed samples were characterized, and the tension-tension fatigue properties of the samples were evaluated via experimentation. The results provided preliminary information about the effect of electropolishing on surface crack initiation of the electron beam melted Ti6Al4V parts.

Keywords: Surface treatment, electropolishing, electron beam melting, Ti6Al4V, fatigue

## **Introduction**

Powder bed fusion additive manufacturing (AM) processes have been widely used for direct fabrication of metal structural components that are used in various industries such as aerospace, biomedicine and automobile. It is expected that AM could bring about significant performance improvement and cost reductions for aerospace component manufacturing [1, 2], and various aerospace companies such as GE Aviation have already started to mass produce AM components in structurally non-critical parts [3, 4]. However, use of metal AM components in structurally critical parts is still limited for short-term use and demonstrations [5-7]. This is partly caused by the relatively low fatigue life of AM metal components as results of both internal and surface defects [8, 9]. Surface defects are intrinsic to powder bed fusion AM processes, which is mainly caused by the partial sintering/melting of the powder particles at the surfaces of the parts due to heat dissipation effects as shown in Fig.1. These surface imperfections often become crack initiation sites upon cyclic loading, which significantly impair the fatigue strength of the structures. It has been suggested that surface defects might be one of the major contributors of the low fatigue strength of metal parts fabricated by powder bed fusion AM processes. [8, 10, 11]. For Ti6Al4V this issue could be further aggravated due to the high notch sensitivity of the material.

Compared to laser melting processes, electron beam melting (EBM) processes usually exhibit higher surface roughness due to higher processing temperature, larger powder particle sizes and larger energy beam dimensions. The surface roughness of the EBM parts spread over a broad range but could often exceeds 30 $\mu$ m depending on various factors such as process parameters, material types, powder characteristics and surface orientations [15, 16]. As a result, the typical fatigue endurance of EBM parts are often only about 10% of that of wrought parts when the surface is not treated [9]. In order to achieve satisfactory long-term performance, surface treatment is necessary.

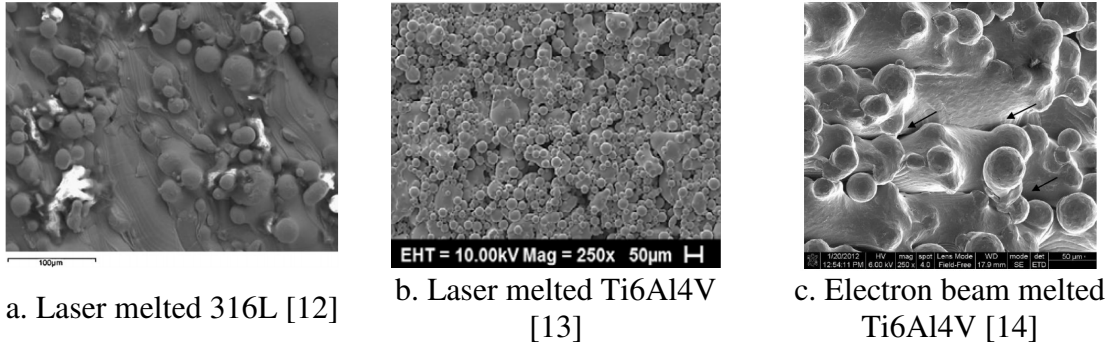


Fig.1 Surface finish of metal powder bed fusion AM parts

In previous studies, the authors investigated the feasibility of utilizing electropolishing for the direct treatment of Ti6Al4V parts fabricated by EBM [17, 18]. Through limited amount of experimentation, it was found that the electropolishing process has the potential to significantly improve the surface quality of the EBM Ti6Al4V parts, although the surface polishing effects exhibit rather large variability, and the experimental conditions were rather difficult to repeat accurately. Additional design efforts were dedicated to the improvement of the experimental devices and the setup of experimentation in the attempt to reduce the random errors of the process. In addition, based on the established electropolishing parameters, fatigue testing samples were polished and tested in order to evaluate the effect of surface treatment on the fatigue performance improvement. This paper will present these works in details.

### **Electropolishing surface treatment: Theories**

Electropolishing process is an electrochemical process in which chemical corrosion of the anode through electrolyte takes place with the assistance of electrical currents. Traditionally electropolishing process is mostly applied on pre-treated surfaces to achieve further improvement of surface finish. Electropolishing effect can be categorized into macromachining and micromachining, which are driven by different mechanisms. As shown in Fig.2, macromachining, or anodic leveling, is effective in removing macroscopic surface features such as surface waviness, and micromachining, or anodic brightening, is effective in removing smaller surface features (<1µm) and achieving mirror-like surface finish [19]. Both types of electropolishing are largely controlled by the mass transportation processes in the electrolyte, which in turn is often driven by the combined effect of surface diffusion barrier layer formation and surface ionic diffusion.

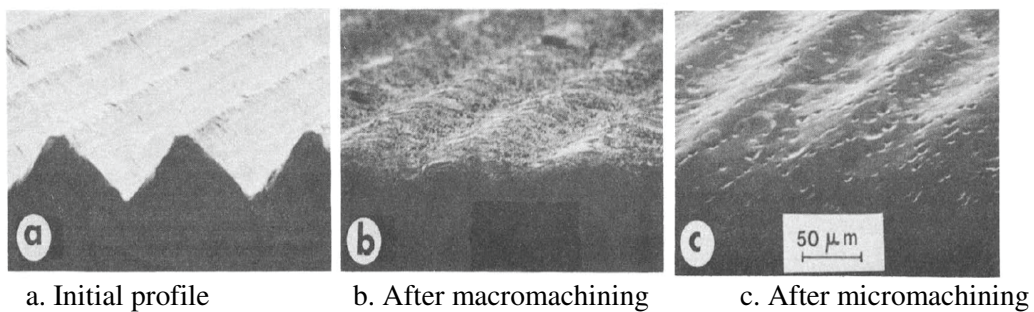


Fig.2 Progression of electropolishing [19]

During the electropolishing process, the electrolyte regions near the anode surface will accumulate higher levels of ionic reaction products, which potentially serve as diffusion barriers for further reactions. In addition, anodic passivation caused by the formation of surface metal oxide films could potentially introduce additional diffusion barriers [20]. The diffusion barrier layer inhibits cationic diffusion from the anode and therefore limits the electropolishing rate.

The dynamic balance during the electropolishing process determines the thickness and characteristics of the diffusion barrier layer. As shown in Fig.3a, when no fluid flow is present, the profile of the diffusion layer would be hypothetically determined by the profile of the anode surface driven by the diffusion laws. However, due to the presence of electrolyte flow in reality, the actual profile of the surface tend to become thicker towards the recessing features (e.g. valleys) on the surfaces as shown in Fig.3b. As a result, the cationic diffusion in these areas are more inhibited compared to the protruding areas, which consequently leads to gradual leveling of the surface [21]. The electrolyte flow can be driven either by diffusion and convection or by external agitation, which makes it possible to adjust the surface diffusion layer profile during the electropolishing process. This unique characteristic makes electropolishing promising in removing surface features at different scales and improving surface finish for AM metal parts.

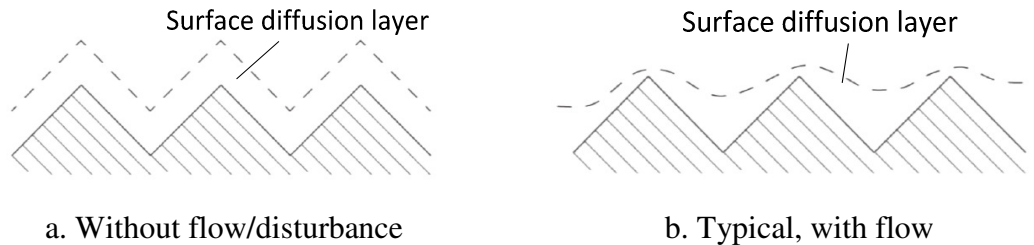


Fig.3 Diffusion layer profile in electropolishing [21]

Typically the ideal formation of the surface diffusion layers occur within certain process windows. Under a certain temperature and experimental setup, the voltage-current curve of the electropolishing often exhibits the characteristic shown in Fig.4. A plateau stage exists at medium range of voltage levels, which correspond to the optimal electropolishing window in which dynamic balance is achieved for the surface diffusion layer [19-21]. Very high voltage potentially results in the breakdown of the surface diffusion layer and over-polishing which is termed pitting, while very low voltage might be inadequate to drive the reactions. As mass transportation processes are strongly influenced by temperatures, the optimal processing window for electropolishing might alter at different temperatures, which must be carefully monitored.

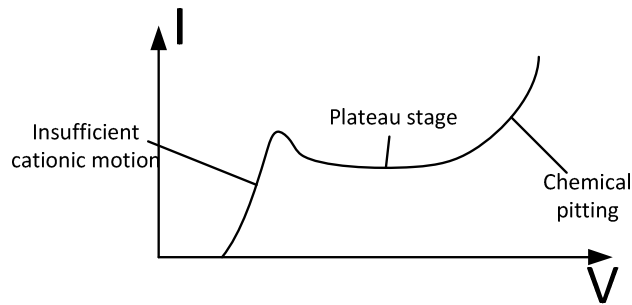
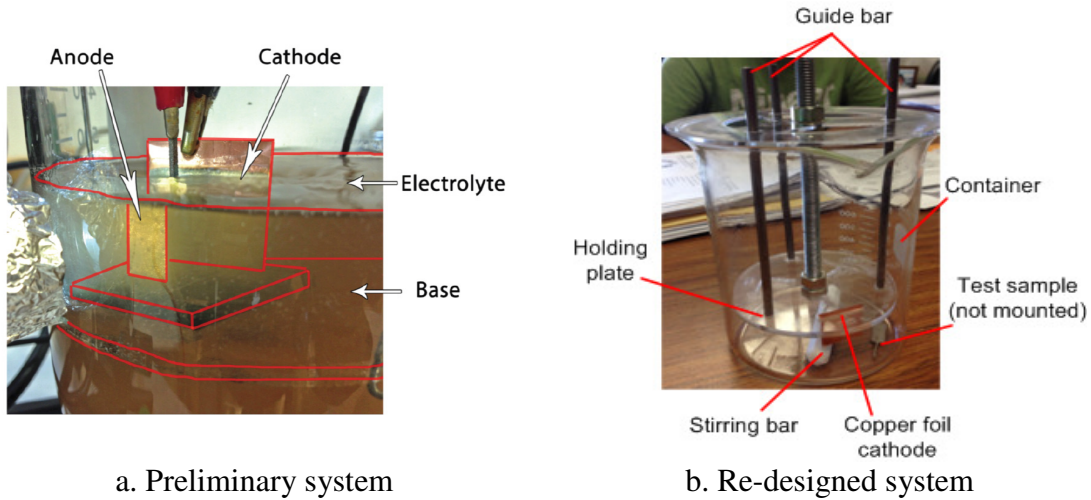


Fig.4 Typical electropolishing voltage-current curve

Previous literatures of electropolishing with Ti6Al4V were mostly carried out with highly hazardous water-based electrolytes such as hydrofluoric acid and perchloric acid in order to overcome the significant oxide formation issue as a result of the reactivity of titanium [22-24]. Recently, non-aqueous electrolyte based on ethyl alcohol and isopropyl alcohol was reported to be successfully used for Ti6Al4V electropolishing [25]. Due to the lack of water, the oxide film formation could be significantly reduced, which might have contributed to the absence of plateau stage in the voltage-current curve. As a result, a moderate level of voltage was determined as the processing parameter, which generated 6-8kA/m<sup>2</sup> of current density during the electropolishing. In previous studies by the authors, the same electrolyte recipe was followed, and the processing voltage was also determined in a similar manner without the observation of apparent plateau stage in the voltage-current curve [17, 18].

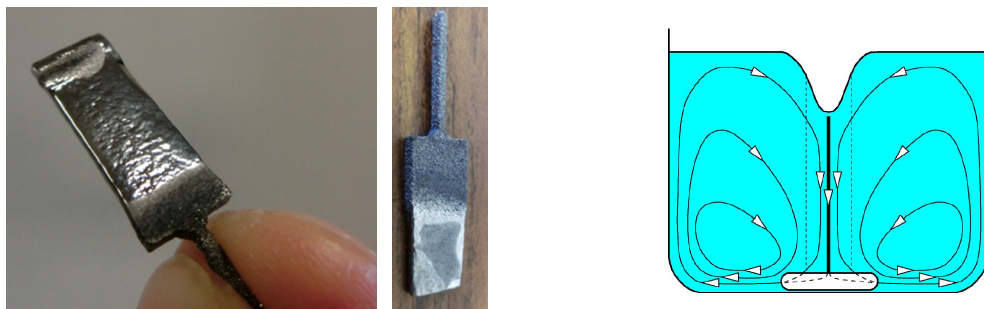
### **Experimental setup**

Multiple iterations were taken in the design of the electropolishing systems. While a rather rudimentary system as shown in Fig.5a was used during the early feasibility study, it was quickly decided that such setup lacks adequate basic control with the processes. In this setup, the electrodes were held by a plastic base plate that was submerged in the electrolyte, and a stirring bar was used to introduce fluid stirring effect in order to promote electropolishing reaction. Due to the concentric flow pattern introduced by the stirring mechanism, the placement of the samples in the middle of the container was considered to be rather ineffective in ensuring consistent electrolyte refreshing at the anode surfaces. In addition, it was also observed that the plastic substrate deteriorated quickly under the severe processing conditions. Therefore, a re-design was devised as shown in Fig.5b, in which the fixation of the electrodes was realized upside down through a plastic holding plate, and the anode was placed towards the edge of the container, which allows it to align better with the electrolyte flow pattern. The holding plate was designed so that multiple configurations for electrode distance could be selected.



a. Preliminary system  
 b. Re-designed system  
 Fig.5 Stirring bar assisted system design

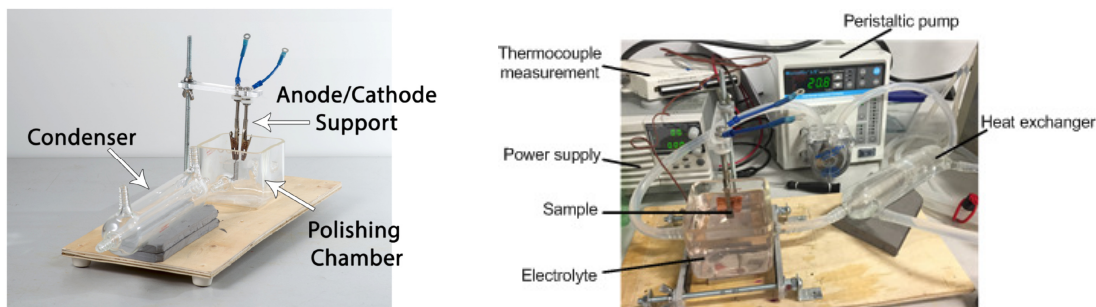
The redesign as shown in Fig.5b was used to study the relationships between various electropolishing parameters including voltage, temperature, electrode spacing and polishing time on the final surface quality of the EBMTi6Al4V samples. Promising results of surface roughness lower than 100nm Ra was realized, although it was also found that the sample were polished at significantly different rates in different locations as shown in Fig.6a [17]. This was concluded to be caused mostly by the irregular flow pattern of the electrolyte using stirring mechanism. In fact, sufficient rotation-induced bottom stirring introduces vortex flow in the liquid in a beaker, which exhibits axially symmetric flow that circulates from the bottom center towards the bottom edge and then ascend to the upper surface of the beaker before flowing down along the central axis again, as shown in Fig.6b. In addition, due to the exothermal reaction that occurred during the electropolishing process, the electrolyte needed to be constantly cooled by placing the beaker in an ice-water bath, which became insufficient when high voltage was used for the electropolishing.



a. Sample [17]  
 b. Stirring induced vortex flow [25]  
 Fig.6 Non-uniform electropolishing via stirring mechanism

In order to further improve the electrolyte flow uniformity, a new circulating flow control system was designed. Fluid pump driven flow control was introduced. In the selection of pump mechanism, rather stringent material restrictions were imposed by the electropolishing chemicals. Due to the use of strong oxidizing salts (zinc chloride and aluminum chloride), organic solvent (alcohols), and the existence of electrical voltage, the material used as fluid channel must be both chemically inert and electrically insulating. As

shown in Fig.7a, a rectangular-shaped glass container with an inlet and outlet in opposite sides was custom made and used as the electropolishing container. A Liebig condenser heat exchanger was used as the cooling mechanism, and ice water or water with either controlled temperature was used as the cooling media. The circulation of the electrolyte was driven by a peristaltic pump, which allows for isolation of fluid from the pump components in order to avoid corrosion. Silicone fluid tubing was used due to its good chemical resistance to both alcohols and oxidizing salts.



a. Container and heat exchanger

b. Circulation system

Fig.7 Electropolishing device with pump driven circulation

It was soon realized that the cooling efficiency of such setup was still insufficient to cool the electrolyte once the electropolishing process initiated. In order for the Liebig condenser to become efficient, large heat exchange contact areas and longer contact time are desired. However, in the current study, due to the need of higher flow rate for efficient electropolishing, the heat exchange time is rather limited. The rather short heat exchange contact length of the Liebig condenser was determined to be insufficient, and a replacement Graham condenser with helical coil was implemented. The latest version of the electropolishing system is shown in Fig.8.

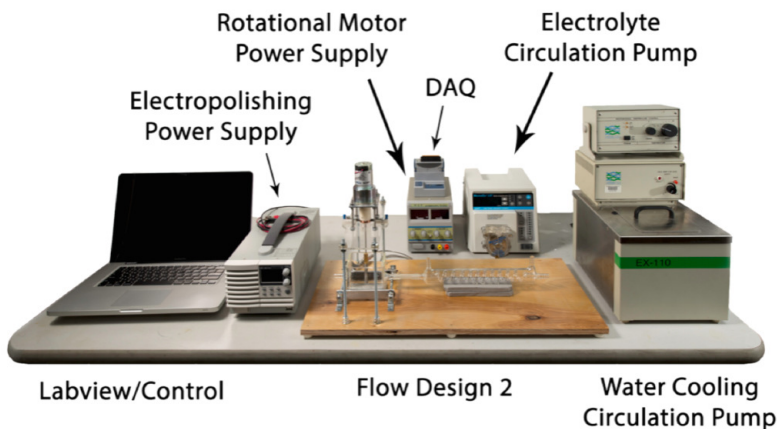


Fig.8 Electropolishing system with Graham condenser

### Electropolishing experiments

In this study the electrolyte was made by following the recipe provided in previous literatures: 1L of electrolyte should consist of: 700mL of ethyl alcohol, 300mL of isopropyl

alcohol, 60g AlCl<sub>3</sub> and 250g ZnCl<sub>2</sub> [26]. All the chemicals used for the experiments are anhydrous. The DC voltage-current curves of different electropolishing system setups were evaluated at room temperatures for multiple electrode spacing setting. As shown in Fig.9, no obvious plateau stage was observed, which agrees with the previous study with this type of electrolyte [26], which was attributed to the lack of formation of the oxide films. For the system with pump driven flow circulation, 60-80V was selected to be the operational voltage range, which correspond to about 6-8kA/m<sup>2</sup> of current densities for the samples used for this study. This range was considerably higher than the values used in previous studies [26].

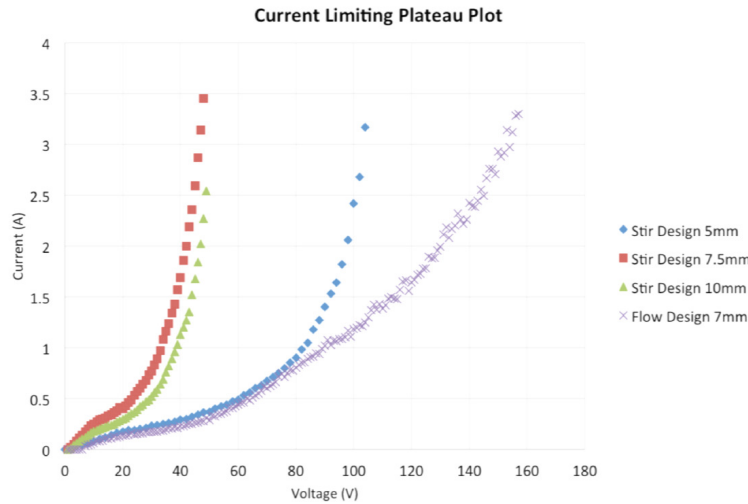


Fig.9 Voltage-current responses of different electropolishing systems

Preliminary works determined that the electrode spacing is less critical in the polishing process [17]. In addition, smaller electrode spacing might reduce the fluid flow between the two electrodes and therefore affect the electropolishing efficiency. As a result, for the consequent experiments, electrode spacing of approximately 7.5-8mm was used without further modification.

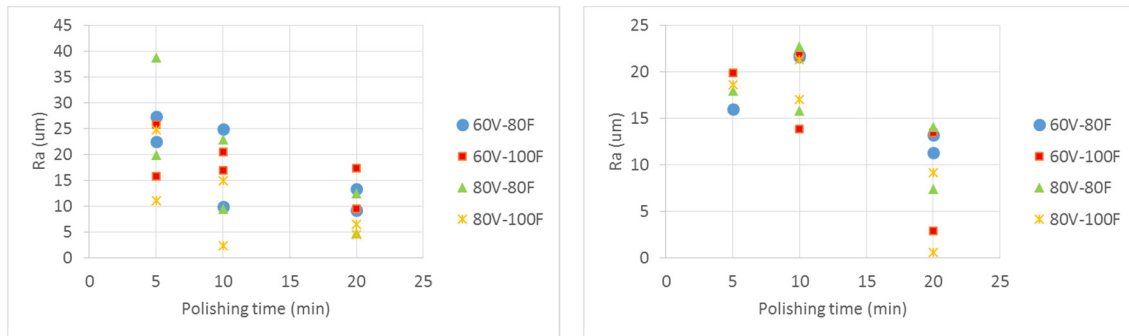
A full factorial experimental design was carried out with three input variables: voltage, temperature and polishing time. The levels of each input variables are shown in Table 1. Among the three input variables the temperature is the most difficult one to control. Therefore, during the experiment intermittent polishing was used, which allowed additional time for the electrolyte to cool down.

Input variable	Levels
Voltage (V)	60, 80
Temperature (°F)	80, 100
Polishing time (min)	5, 10, 20

Table 1 Input variables for electropolishing experimental design

Samples with flat plate design were fabricated in Arcam EBM S400 system with Ti6Al4V using the system default parameter setting. All the samples were oriented in such a way that the surfaces to be polished are aligned vertically in the building chamber,

which results in a rather rough surface finish. The surface roughness of the as-received samples were measured via the Dektak 8 profilometer, and the results showed a roughness of 30-40 $\mu\text{m}$ . It was suspected that some of the samples were rougher due to the vacuum issue with the EBM system. Two samples were polished for each electropolishing parameter setup. After electropolishing, the surface roughness of both side of the samples were measured using the same method, and the results are shown in Fig.10. In general the most significant factor appears to be the polishing time since a broad range of polishing time was investigated. Non-uniform polishing was observed despite the improved electrolyte flow control, which was likely due to the non-uniform interaction between electrolyte and the samples at different locations. As a consequence, the front side and the back side of the samples were polished differently. The surface roughness of the samples after polishing for 20 minutes were approximately 5-15 $\mu\text{m}$  for the front sides and 0-15 $\mu\text{m}$  at the back sides. The combination of voltage and temperature did not appear to have as significant effects, although the 80V-80F and 60V-100F combinations showed slightly better surface roughness consistency. This was intuitive since that both higher voltage and higher temperature would promote the electropolishing rates, while their combination might introduce excessive polishing effects.



a. Front side  
b. Back side  
Fig.10 Surface roughness of electropolished samples

As one of the benefits of interest of electropolishing is the fatigue performance of the EBM parts, additional experiments were carried out with both 80V-80F and 60V-100F parameter combinations to evaluate the impact of electropolishing on the fatigue endurance of the samples. Fatigue samples as shown in Fig.11 were designed and fabricated using the same EBM parameter setting that was used for the samples for the polishing study. The fabricated samples were also shown in Fig.11. An additional feature was designed to facilitate the gripping of the samples during electropolishing, which could be subsequently removed without affecting the part quality.

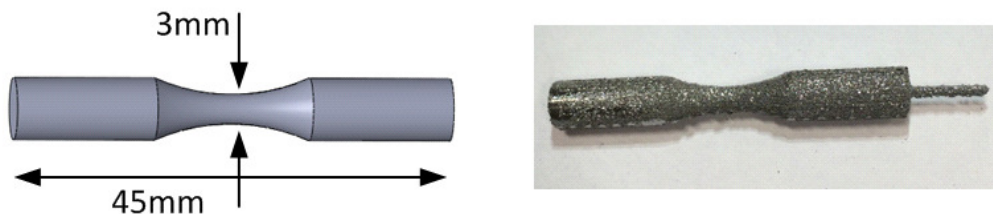


Fig.11 Fatigue sample



Multiple samples were polished for both the 80V-80F and 60V-100F parameter setting. In order to maximize the polishing effects, all the samples were polished for 20 minutes. Due to the rotationally symmetric feature of the fatigue sample, during the electropolishing process the samples were rotated four times with 90° of rotation angle in the attempt to achieve more uniform polishing around the entire parts. The samples were then machined to create smooth gripping surface. Fig.12 shows some the polished samples after machining. The samples were tested in an Instron Electroplus E10000 fatigue tester under tensile-tensile cyclic loading.



Fig.12 Electropolished fatigue samples after machining

Before the fatigue testing of the electropolished samples, the as-received samples were tested under different loading ratios  $R$  in order to establish preliminary ideas with the fatigue performance of the control group. After evaluation it was found that the as-received samples exhibit drastic reduction of fatigue life when the  $R$  ratio was between 0.25 and 0.3. In addition, it was found from the fracture surface analysis that for samples tested with  $R=0.25$  the prevailing failure mechanism was surface cracking, while for samples tested with  $R=0.3$  internal defect cracking appeared to be more dominant. As a result, loading ratio of  $R=0.25$  was determined to be used for all the electropolished samples in order to evaluate the effectiveness of the surface treatment process in reducing surface cracking under cyclic loading. The results are shown in Fig.13. Although limited data points were available, the fatigue life of multiple electropolished samples achieved over 1.6 million, which correspond to over 100% improvement compared to the as-received samples (~450000). It must be noted that most fatigue testing for the electropolished samples stopped after about 1.6 million due to the time constraints, and only one sample polished by 60V-100F parameter combination was tested to 3.3 million cycles without part failure. Therefore, it could be expected that the beneficial effect of electropolishing surface treatment could be even more significant. On the other hand, the fatigue strength of the electropolished samples appeared to scatter more significantly compared to the as-received samples. Further investigation will be carried out to further identify such effect, however it was speculated that this was partly caused by the non-uniform electropolishing and the formation of new surface defects.

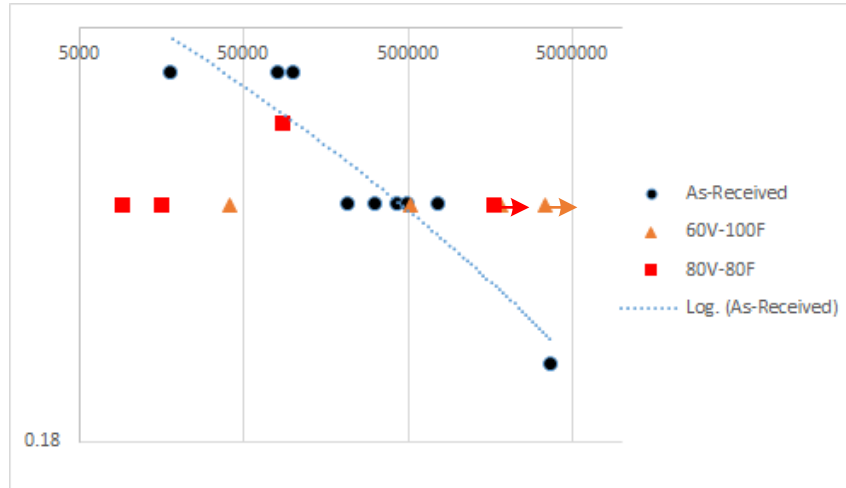


Fig.13 Fatigue properties of the electropolished samples

### Conclusions

In this project multiple electropolishing systems were designed and evaluated. It was found that electrolyte flow control and temperature control were the two most significant process control variables. Circulating flow driven by peristaltic pump provides protection to the pump and improved flow control with the electrolyte. In addition, a Graham condenser heat exchanger was also implemented in order to improve the cooling of the electropolishing process.

Preliminary mechanical testing showed impressive results, with fatigue life increased by over 300% for multiple samples polished at both 80V-80F and 60V-100F processing parameter combinations for 20 minutes. Although the sample size was relatively limited, the preliminary observations showed promising potential of the electropolishing process in improving the mechanical properties of Ti6Al4V parts fabricated by EBM and other powder bed fusion AM processes.

### Acknowledgement

This work was partially supported by NASA Marshall Space Flight Center Contract #SC-2019-004 and NASA Kentucky EPSCoR Grant RID-3 #7800003125. The authors would like to acknowledge the support of Rapid Prototyping Center (RPC) and Conn Center of Renewable Energy at University of Louisville.

### Reference

- [1] D. Godfrey. Additive manufacturing (AM) and the Honeywell global initiative. Proceedings of the International Society of Air Breathing Engines Conference, Phoenix, AZ, 2015.
- [2] GE Global Research. <http://www.geglobalresearch.com/innovation/3d-printing-creates-new-parts-aircraft-engines>. Accessed July 2016.
- [3] GE Reports. <http://www.gereports.com/post/116402870270/the-faa-cleared-the-first-3d-printed-part-to-fly/>. Accessed July 2016.

- [4] Fortune report. <http://fortune.com/2015/03/05/ge-engine-3d-printing/>. Accessed July 2016.
- [5] NASA news. <http://www.nasa.gov/centers/marshall/news/news/releases/2015/piece-by-piece-nasa-team-moves-closer-to-building-a-3-d-printed-rocket-engine.html>. Accessed July 2016.
- [6] Aerojet Rocketdyne. <http://www.rocket.com/article/setting-standard-3-d-printed-rocket-engines>. Accessed July 2016.
- [7] Monash Centre for Additive Manufacturing. [https://platforms.monash.edu/mcam/index.php?option=com\\_content&view=category&layout=blog&id=18&Itemid=195](https://platforms.monash.edu/mcam/index.php?option=com_content&view=category&layout=blog&id=18&Itemid=195). Accessed July 2015.
- [8] H. Gong, K. Rafi, T. Starr, B. Stucker. Effect of defects on fatigue tests of as-built Ti-6Al-4V parts fabricated by selective laser melting. Proceedings of the Solid Freeform Fabrication (SFF) Symposium, Austin, TX, USA, 2012.
- [9] P. Li, D. H. Warner, A. Fatemi, N. Phan. Critical assessment of the fatigue performance of additively manufactured Ti-6Al-4V and perspective for future research. International Journal of Fatigue. 85(2016): 130-143.
- [10] N. W. Hrabe, P. Heintl, B. Flinn, C. Korner, R. K. Bordia. Compression-compression fatigue of selective electron beam melted cellular titanium (Ti-6Al-4V). Journal of Biomedical Materials Research, Part B- Applied Biomaterials. 99(2011): 313-320.
- [11] S. A. Yavari, R. Wauthle, J. van der Stok, A. C. Riemsagel, M. Janssen, M. Mulier, J. P. Kruth, J. Schrooten, H. Weinans, A. A. Zadpoor. Fatigue behavior of porous biomaterials manufactured using selective laser melting. Materials Science and Engineering C. 33(2013); 4849-4858.
- [12] G. Strano, L. Hao, R. M. Everson, K. E. Evans. Surface roughness analysis, modelling and prediction in selective laser melting. Journal of Materials Processing Technology. 213(2013): 589-597.
- [13] J. Vaithilingam, R. D. Goodridge, S. D. Christie, S. Edmondson, R. J. M. Hague. Surface modification of selective laser melted structures using self-assembled monolayers for biomedical applications. Polishing. Proceedings of Solid Freeform Fabrication (SFF) Symposium, Austin, TX, 2012.
- [14] K. Rafi, H. Karthik N.V, T. L. Starr, B. E. Stucker. Mechanical property evaluation of Ti-6Al-4V parts made using electron beam melting. Proceedings of Solid Freeform Fabrication (SFF) Symposium, Austin, TX, 2012.
- [15] A. Safdar. Microstructures and surface roughness of EBM produced Ti-6Al-4V. PhD Dissertation. University of Malmo, Malmo, Sweden, 2010.
- [16] J. Karlsson. Optimization of electron beam melting for production of small components in biocompatible titanium grades. Uppsala University, Uppsala, Sweden, 2015.
- [17] L. Yang, H. Gu, A. Lassell. Surface treatment of Ti6Al4V parts made by powder bed fusion additive manufacturing processes using electropolishing. Proceedings of Solid Freeform Fabrication (SFF) Symposium, Austin, TX, 2014.

- [18] L. Yang, A. Lassell, G. P. V. Paiva. Further study of the electropolishing of Ti6Al4V parts made via electron beam melting. Proceedings of Solid Freeform Fabrication (SFF) Symposium, Austin, TX, 2015.
- [19] D. Landolt. Fundamental aspects of electropolishing. *Electrochimica Acta*. 32(1987), 1:1-11.
- [20] T. P. Hoar, D. C. Mears, G. P. Rothwell. The relationships between anodic passivity, brightening and pitting. *Corrosion Science*. 5(1965): 279-289.
- [21] C. Clerc, M. Datta, D. Landolt. On the theory of anodic leveling: model experiments with triangular nickel profiles in chloride solution. *Electrochimica Acta*. 29(1984), 10: 1477-1486.
- [22] J. Pelleg. Electropolishing of titanium. *Metallography*. 7(1974): 357-360.
- [23] J. B. Mathieu, H. J. Mathieu, D. Landolt. Electropolishing of titanium in perchloric acid-acetic acid solution. I. Auger electron spectroscopy study of anodic films. *Journal of Electrochemical Society*. 125(1978), 7: 1039-1043.
- [24] J. B. Mathieu, D. Landolt. Electropolishing of titanium in perchloric acid-acetic acid solution. II. Polarization behavior and stoichiometry. *Journal of Electrochemical Society*. 125(1978), 7: 1044-1049.
- [25] G. Halasz, B. Gyure, I. M. Janosi, K. G. Szabo, T. Tel. Vortex flow generated by a magnetic stirrer. *American Journal of Physics*. 75(2007), 12: 1092-1098.
- [26] K. Tajima, M. Hironaka, K.-K. Chen, Y. Nagamatsu, H. Kakigawa, Y. Kozono. Electropolishing of CP titanium and its alloys in an alcoholic solution-based electrolyte. *Dental Materials Journal*. 27(2008), 2: 258-265.