

SMASIS2017-3873

A COMPARATIVE STUDY OF SURFACE CLEANING TREATMENTS FOR 3D PRINTED MEDICAL IMPLANTS

Lucy Guo, Zhiqiang Xie, Hong Yao and Ying Wang*

Department of Mechanical & Industrial Engineering,

Louisiana State University, Baton Rouge, LA 70803, USA.

*Corresponding author: Prof. Ying Wang, E-mail: ywang@lsu.edu.

ABSTRACT

In the field of Additive Manufacturing (AM), one of the major applications of laser-based 3D metal printing is the creation of custom implants for medical purposes. However, a significant challenge in the manufacturing of implants using Selective Laser Melting (SLM) is the formation of partially melted particles on the surface of medical implants. These particles result in a multitude of issues including plurality of structurally weak points on the designed implants, obstruction of important design features, and possibility of dislodgement over the service life span, thereby posing a threat to the recipient. To address the above challenges, it is imperative to develop a simple but effective surface cleaning method to remove partially melted particles from the surface without damage to the designed medical implants.

In this work, a comparative study was conducted to investigate the effect of both chemical and electro-plasma based cleaning processes on the removal of partially melted particles from the surfaces of 3D printed Ti-6Al-4V medical screw implants. These techniques include chemically polishing implants with HF-HNO₃ acid solutions and using an electro-plasma based cleaning process. With the field of additive manufacturing rapidly expanding, this work offers valuable insight on proper post-process treatment of 3D printed parts for future medical purposes in biomedical fields.

INTRODUCTION

Selective laser based additive manufacturing is a principal technology capable of creating complex and unique 3D metallic parts with a wide range of applicability [1-2]. By employing a high power laser source and fine metallic powders, SLM

creates 3D metal parts using computer aided design (CAD)-directed, layer-by layer selective melting [1,3]. 3D metal printing has vast potential within the medical field, particularly in the production of custom implants [4]. However, many medical institutions are deterred from utilizing this method because of the partially melted particles that form on the surface of medical implants during SLM. As stated before, these partially melted powder particles result in plurality of structurally weak locations throughout the overall implant configuration and can even dislodge from the surface of these implants, resulting in possibly harm to the recipient. Furthermore, for specific purposes such as osseointegration, partially melted surface particles obstruct necessary design feature on implants, thus limiting their performance [5]. With the rising popularity of AM in medical disciplines such as biomedical technology and tissue engineering, a proper procedure for post-processing of SLM printed parts in medical implants is essential.

Current surface cleaning methods of additively manufactured metal parts mainly include sandblasting, ultrasonic nanocrystal surface modification (UNSM), electrochemical polishing and hand finishing. However, the above cleaning procedures are insufficient for the proper removal of partially melted powders on the surface of 3D printed medical implants with complex geometry and smaller overall dimensions (common characteristics of many medical implants) [6-8].

Among the few metal alloys suitable as materials for medical implants, titanium alloys (mainly Ti-6Al-4V) are the primary choice for dental implants and are also clinically used in artificial bone and tissue implants due to their properties in

biocompatibility, corrosion resistance, strength, etc. [9] In this paper, post-processing procedures for 3D printed Ti-6Al-4V medical screw implants were studied. Although cleaning methods using various chemicals such as hydrofluoric acid (HF) or hydrofluoric/nitric acid (HF/HNO₃) solutions have been analyzed before for titanium scaffolds [10], as well as surface modification through cathodic electrolytic plasma cleaning on varied metal surfaces [11], comparisons on the efficiency of these two processes on 3D printed titanium medical implants have not been reported.

With the support of Louisiana Consortium for Innovation in Manufacturing and Materials (CIMM) team, during the first stage of the experiments, custom medical implants were designed using the 3D CAD software Solidworks and several other printer specific software packages (Figure 1). These parts were then fabricated from Ti-6Al-4V powders under certain operation procedures and parameter settings for optimal structural integrity using a ConceptLaser Mlab system in CIMM Core User Facility. Once printed, an investigation on the effects of both chemical polishing and electroplastic polishing on the morphology and surface consistency of the samples was conducted. The goal of this study is to compare chemical and electro-plasma based cleaning methods and identify an effective post-process treatment procedure to achieve the most uniform surface cleaning on 3D printed titanium alloy parts.

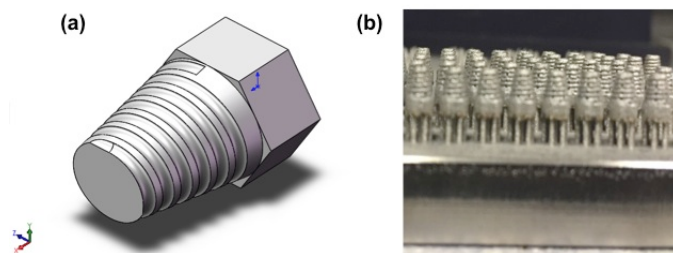


Figure 1. (a) Solidworks medical screw design and (b) 3D printed samples.

METHODOLOGY

Medical Screw Modeling and Fabrication

The 3D model of the titanium screws was created in the computer-aided design software Solidworks. To generate the 3D model, a hexagonal base of height 1.50 mm and diameter 3.00 mm was first drawn. A cylinder of 3.00 mm base diameter, 3.00 mm length and a taper of 9.46 degree was extruded normal to the hexagonal base. A helix and spiral framework of height 2.80 mm and 6 revolutions was added encompassing the axial surface of the tapered cylinder and a cut sweep of 1/4 th of a 0.30 mm circle was used along this framework to create the threads of the samples. Pores of sizes 0 μm , 100 μm , 200 μm and 300 μm were added along the sides and ends of the screws (Figure 2).

Four types of specimens were fabricated, as shown in Fig. 2, using Ti-6Al-4V powder on a ConceptLaser Mlab-cusing-R SLM machine. Titanium screws were printed in an argon inert atmosphere where oxygen values were kept in between 0.3-0.5%. ConceptLaser Mlab utilizes a Yb-Fibre laser which has a laser power of up to 100 W, a maximum scanning speed of 7000 mm/s, and a focus diameter of 50 μm . A constant layer thickness of 25 μm was also chosen for the production of these samples. During this fabrication process, support structures were generated on top hexagonal base for easy separation from the building block (Fig.1 right).

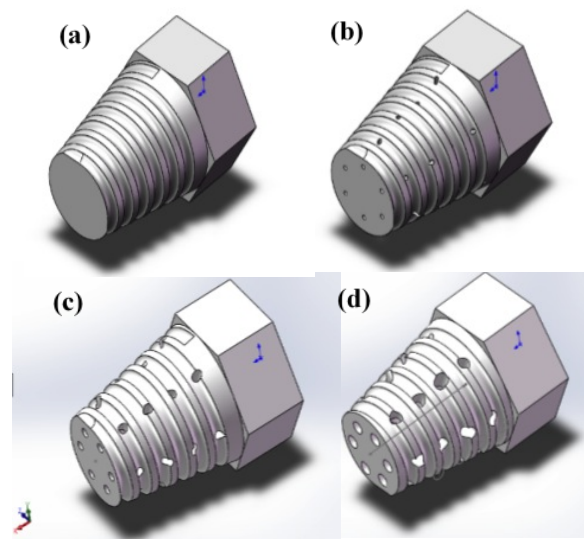


Figure 2. Four types of medical screw specimens with pore sizes of (a) 0 μm , (b) 100 μm , (c) 200 μm , (d) 300 μm .

Chemical Polishing

Chemical polishing was conducted in a hydrofluoric/nitric acid mixture of concentrations 2.2% and 20% respectively. In specific, to make 50 mL of acid mixture, 2.29 mL of HF and 15.39 mL of HNO₃ were added into 32.32 mL of water. Tests were conducted on nonporous screw specimens as well as specimens with engineered pores. After submersion in separate beakers for 3, 6 and 9 minutes, respectively, each sample was rinsed three times with distilled water, two times with ethyl alcohol, and then cleaned using an ultrasonic cleaner. Afterwards screws were left out to dry completely. The mass of the samples was measured before and after chemical polishing, and scanning electron microscopic (SEM) characterization was carried out to observe surface morphology change after chemical polishing treatment.

Electro-Plasma Surface Cleaning

For electro-plasma cleaning, a simple setup was derived consisting of a power source, a sacrificial electrode, and the work piece (medical screws). Sodium bicarbonate, a safe and cost effective chemical compound was used as the electrolyte.

The work piece was connected to the negative side of the power source and the sacrificial scrap metal was connected to the positive side of the power source (Fig. 3). Both pieces were placed in a 1000 mL beaker containing 600 mL of water and 45g of sodium bicarbonate for optimal concentration of electrolyte according to a previous study conducted [11].

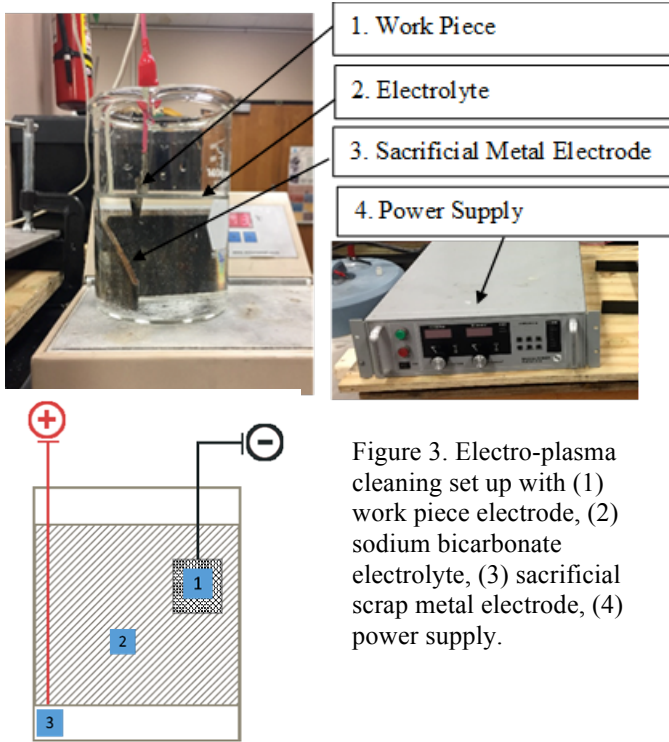


Figure 3. Electro-plasma cleaning set up with (1) work piece electrode, (2) sodium bicarbonate electrolyte, (3) sacrificial scrap metal electrode, (4) power supply.

When a current is applied to this setup at a low voltage, common processes such as anodizing and electroplating take place. As the voltage increases, gas evolution becomes very rapid and micro-arc discharge begins to occur, leading to a plasma envelope around the work piece [12]. To observe this process, specimens were first treated at 100 V for 20, 30, and 45 seconds in coordination with electroplating methods. Then the printed screws were treated under higher voltages of 140V and 160V, which are voltages within the optimal region for the formation of an electro-plasmic envelope, for 3 seconds each to observe any noticeable differences with increasing voltage and decreasing treatment time.

RESULTS AND DISCUSSION

Chemical Polishing

The fabricated medical screws were chemically polished using HF/HNO₃ mixed acid solutions. The polishing time was 3, 6, and 9 minutes. The mass variations after chemically polishing treatment in the HF/HNO₃ solutions are shown in Figure 4. Independent of pore size, an increase of mass change is observed when the soaking time was extended from 3 minutes to 6 minutes. However, mass change for the other medical screws of pore size 100 μm , 200 μm , and 300 μm did

not vary as drastically. This observation probably indicates that the effectiveness of chemical treatment after 6 minutes declines, and the optimal treatment time for 3D printed titanium parts is around 6 minutes.

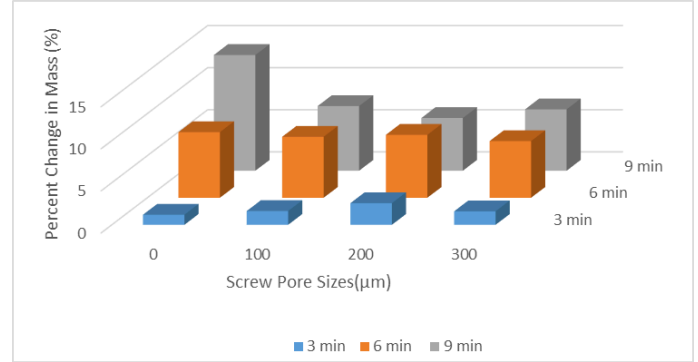


Figure 4. Mass changes of medical screws after chemical polishing in mixed HF/HNO₃ acids with treatment time of 3, 6 and 9 min, respectively.

Figure 5a-h shows the SEM images of non-porous 3D printed screws after chemical polishing. As shown in Figure 6a and b, one can clearly observe the partially melted titanium powders on the surface of non-porous 3D printed medical screws. After chemical polishing treatment for 3 minutes, some partially melted titanium particles are removed (Figure 5c and d). With the increase of treatment time to 6 and 9 minutes, partially melted particles are completely removed without damage to the screw framework.

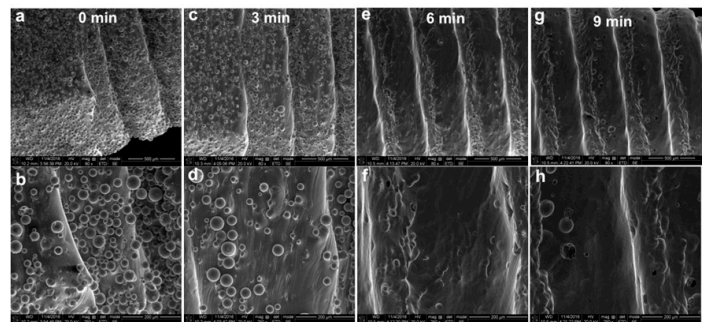


Figure 5. SEM images of non-porous 3D printed screws after chemical polishing in mixed HF/HNO₃ acids with treatment time of (a, b) 0 min, (c, d) 3 min, (e, f) 6 min and (g, h) 9 min, respectively.

SEM images of porous medical screws after chemical polishing are shown in Figure 6a-h. The pores become partially visible with the increase of treatment time to 6 minutes and completely unblocked after 9 minutes of treatment. However, loss of integrity within samples is also observed around the edge of the screw due to chemical polishing treatment (Figure

6g and h). Therefore, by careful comparison of surface morphology change through SEM images and consideration of mass change effectiveness, the optimal time for chemical polishing treatment using HF/HNO₃ mixed acid solution is determined to be 6 minutes, which allows for efficient and uniform removal of partially melted titanium powders without noticeable damage to the overall framework of medical screws.

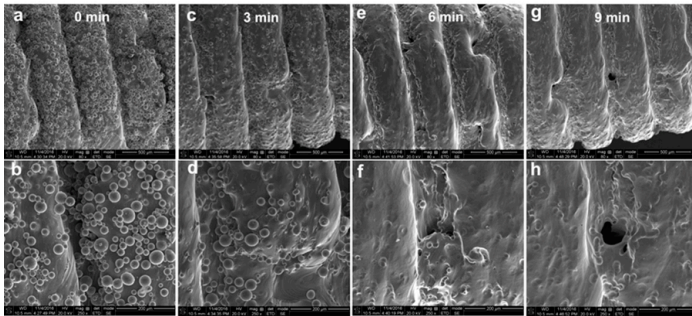


Figure 6. SEM images of porous 3D printed screws with 200µm pore size after chemical polishing in mixed HF/HNO₃ acids with treatment time of (a, b) 0 min, (c, d) 3 min, (e, f) 6 min and (g, h) 9 min, respectively.

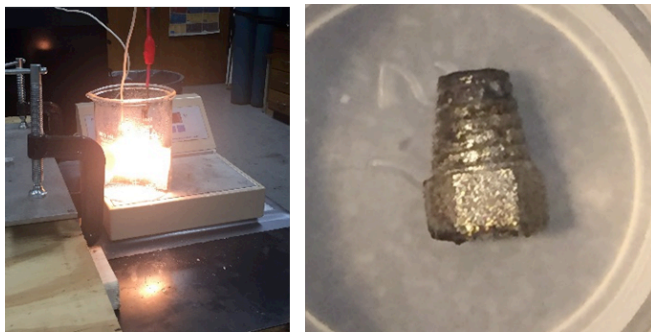


Figure 7. Electrical arc present at beginning of electroplasma cleaning process for 140 V test (left) and uneven cleaning of porous 3D printed screws (right).

Electro-Plasma Surface Cleaning

Electro-plasma cleaning treatments were also conducted for 3D printed medical screws. According to previous studies conducted, during electro-plasma cleaning, high voltages applied along the surface of the work piece can form plasma bubbles, leading to micro-melting and surface morphology change [11-13]. In this study, non-porous screws were electropolished with applied voltage of 100V for 20, 30 and 45 seconds, respectively. After removal of the medical screw from electrolytic bath, an uneven distribution of cleaning on the working piece is observed, which is largely due to the high variability and uncontrollable nature of both the plasma bubbles and electric arc during the surface cleaning process (Figure 7).

Further SEM analysis reveals that a majority of partially melted particles can be removed from the lower end of the non-porous screw after 45 seconds of treatment (Figure 8). In

comparison with chemical treatment, the surface at this area is slightly smoother and the treatment is faster and safer. However, the cleaning only takes place at the very end of the screw where the electric arc is concentrated. In the corresponding tests conducted on porous medical screws at 140V and 160V for 3 seconds, as voltage increases, a general trend of titanium particles removal is observed from Figure 9. The surface, however, is not uniform and the holes within the sample are not fully cleaned due to discrepancies in cleaning areas. Based on the above SEM observations, it is clearly evident that electro-plasma cleaning treatment produces uneven removal of partially melted titanium powders from surfaces of 3D printed screws, and also results in severe damage to the overall framework of medical screws. Therefore, it is not applicable to surface cleaning of 3D printed parts with complex geometry.

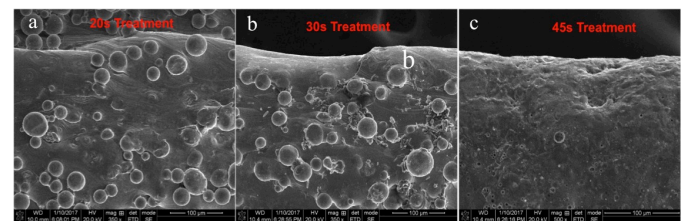


Figure 8. SEM images of porous 3D printed screws after electro-plasma surface cleaning under 100V with treatment time of (a) 20 seconds, (b) 30 seconds and (c) 45 seconds, respectively.

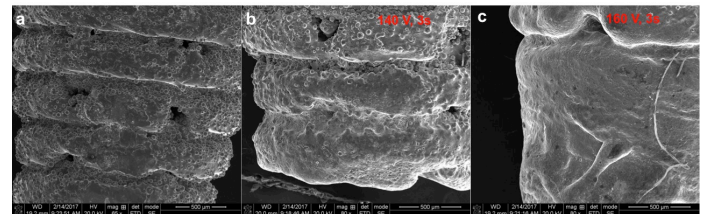


Figure 9. SEM images (a) porous 3D printed screws without treatment, (b) and (c) of 3D printed screws after electro-plasma surface cleaning for 3 seconds under 140 and 160V, respectively.

CONCLUSIONS

In this study, post-processing cleaning procedures for 3D printed Ti-6Al-4V medical screw implants are explored and a comparison between the efficiencies of chemical cleaning treatments and cathodic electrolytic plasma cleaning treatments is provided. Major research findings include: (1) Electro-plasma surface cleaning treatment is both faster and safer than chemical polishing, but it produces uneven removal of partially melted titanium powders from surfaces of 3D printed dental screws, and also results in severe damage to the overall framework of screws. (2) The operation voltage and treatment time play crucial roles in electro-plasma surface cleaning process. As voltage and treatment time increase, a general trend

of melted titanium particles removal from the surfaces of 3D printed screws is observed from scanning electron microscope (SEM). (3) Chemical polishing treatment of 3D printed screws using HF/HNO₃ mixed acid can produce more efficient and uniform removal of partially melted titanium powders. (4) The treatment time for chemical polishing is optimized to ensure efficient and uniform removal of partially melted titanium powders without noticeable damage to the overall framework of 3D printed screws.

With the field of additive manufacturing rapidly expanding, this work offers valuable insight on proper procedures for post processing of 3D printed parts for future medical purposes and may open new routes for future incorporation of additive manufacturing in biomedical fields and exploration of even more effective post processing treatments of 3D printed parts.

ACKNOWLEDGMENTS

This work is funded in part by the NSF EPSCoR CIMM project under award #OIA-1541079 and Louisiana Board of Regents' Supervised Undergraduate Research Experience (SURE) Program. The use of the LSU Shared Instrumentation Facility (SIF), a part of the CIMM Core User Facilities (CUFs), is acknowledged.

REFERENCES

- [1] Santos E.C., Shiomi M., Osakada K., Laoui T., 2006, "Rapid Manufacturing of Metal Components by Laser Forming", *International Journal of Machine Tools and Manufacture*, Vol. 46, Issues 12-13, pp1459-1468.
- [2] Vrancken B., Thijs L., Kruth J., Humbeeck J.V., 2012, "Heat Treatment of Ti6Al4V Produced by Selective Laser Melting: Microstructure and Mechanical Properties", *Journal of Alloys and Compounds*, Vol. 541, pp177-185.
- [3] Olakanmi E., Cochrane R., Dalgarno K., 2015, "A Review on Selective Laser Sintering/Melting (SLS/SLM) of Aluminium Alloy Powders: Processing, Microstructure, and Properties", *Progress in Materials Science*. Vol. 74, pp401-477.
- [4] Lee V.C., 2014, "Medical Applications for 3D Printing: Current and Projected Uses", *P & T: a peer-reviewed journal for formulary*, Vol. 39, pp704-711.
- [5] Tamimi F., Torres J., Al-Abedalla K., Lopez-Cabarcos, Alkhraisat M.H., Bassett D. C., Gbureck U., Barralet J., 2014, "Osseointegration of Dental Implants in 3D-printed Synthetic Onlay Grafts Customized According to Bone Metabolic Activity in Recipient Site", *Biomaterials*, pp5436-5445.
- [6] Duleba B., Greškovič F., Sikora J.W., 2011, "Materials and Finishing Method of DMLS Manufactured Parts", *Transfer inovácií*, Vol. 21, pp143-148.
- [7] Kumbhar N. N., Mulay A. V., 2016, "Post Processing Methods Used to Improve Surface Finish of Products

Which Are Manufactured by Additive Manufacturing Technologies: A Review", *Journal of The Institution of Engineers (India): Series C*, pp1-7.

- [8] Ma C., Dong Y. L., Ye C., "Improving Surface Finish of 3D-Printed Metals by Ultrasonic Nanocrystal Surface Modification", *Procedia CIRP*, Vol. 45, pp319-322.
- [9] Saini M., Singh Y., Arora P., Arora V., Jain K., 2015, "Implant Biomaterials: A Comprehensive Review", *World Journal of Clinical Cases*, Vol. 1, no. 3, pp52-57.
- [10] Łyczkowska E., Szymczyk P., Dybała B., Chlebus E., 2014, "Chemical Polishing of Scaffolds Made of Ti-6Al-7Nb Alloy by Additive Manufacturing", *Archives of Civil and Mechanical Engineering*, Vol. 14, Issue 4, pp586-594.
- [11] Liang J., 2013, "A Study on the Cleaning and Modification of Metal Surfaces by Direct Current Cathodic Electrolytic Plasma Process", Ph.D. Thesis, Louisiana State University, Baton Rouge, LA.
- [12] Legg K., 2008, "Electrolytic Plasma Processing for Sequential Cleaning and Coating Deposition for Cadmium Plating Replacement", Final Report SERDP Project WP-1406, Rowan Technology Group, IL, pp6-10.
- [13] Kumruoglu L.C., Özel A., 2013, "Electrolytic Plasma Surface Cleaning of Industrial Metallic Components", *Proceedings of the 3rd International Congress, Acta Physica Polonica A*, Vol 125, Issue 2, pp379-381.