Materials Research Proceedings 41 (2024) 308-315 https://doi.org/10.21741/9781644903131-35

Improvement of the surface quality of titanium-based design objects produced through WAAM technology using chemical machining: A preliminary study

PERNA Alessia Serena[1,a*], SCHERILLO Fabio[1,b *], SQUILLACE Antonino[1,c], CAMPATELLI Gianni[2,d], CARVALHO H.S.F.L. Gustavo[2,e]

[1]Department of Chemical, Materials and Production Engineering, University of Naples Federico II, Piazzale V. Tecchio 80, 80125 Napoli, Italy

[2] Department of Industrial Engineering - University of Firenze - 50139 Firenze, Italy

[a]alessiaserena.perna@unina.it, [b]fabio.scherillo@unina.it, [c]squillac@unina.it, [d]gianni.campatelli@unifi.it, [e]gustavo.carvalho@unifi.it

Keywords: Additive Manufacturing, WAAM, Chemical Machining, Titanium, Design

Abstract. The quality of the surface is one of the most important factors in the fabrication of a component via additive manufacturing (AM). In particular, when considering the manufacture of workpieces in titanium and its alloys the successful use of surface treatments is essential. In fact, many fracture-related events, in particular fatigue cracks, start near the surface of the component. Numerous techniques based on machining, shot peening, or laser polishing have been proposed to enhance the surface quality. The limitations of these treatments stem from the challenges posed by focusing on complex form components. One of the most promising approaches for achieving homogenous smoothing of intricate objects with internal channels and lattice structure continues to be chemical-based surface treatments. It is a pivotal method to remove material that has been polluted by oxygen during processing. In this instance, the resistance to crack initiation and fracture is fundamentally improved by the removal of a hard, brittle top layer. In this work, HF/HNO_3-based treatment tailored for 3D printed design products is presented.

Introduction

The quality of the surface is one of the most important factors in the fabrication of a component via additive manufacturing (AM) [1]. Surface quality has a significant impact on the mechanical characteristics of the component: sharp peaks and valleys which serve as stress concentrators are typically found on the surface of an additive-manufactured product [2]. In particular, when considering the manufacture of workpieces in titanium and its alloys the successful use of surface treatments is essential [3]. This is due to the fact that many fracture-related events, in particular fatigue cracks, start near the surface of the component [4]. Numerous techniques based on machining, shot peening, or laser polishing have been proposed to enhance the surface quality [5]. Steel has long been subjected to shot peening processes to increase its resistance to the onset of fatigue cracks by applying a compressive force to the surface. Although not novel, the use of this approach to titanium alloy components is more recent than it is to steel [6–8]. Titanium components with stress-free surfaces have a relatively poor resistance to fatigue: the fatigue strength at 10 cycles to failure, expressed as a percentage of the yield stress is typically between 0.4 and 0.5 [9]. This value is further diminished when damage from production procedures like milling is added. Local plastic deformation during shot peening introduces compressive stresses into the material's near-surface region to prevent this additional decrease in fatigue resistance [8].

More recently, it has been demonstrated that applying a laser to induce compressive stress on the surface is quite advantageous [10,11]. This method applies a localized pressure to the material's surface using a high-intensity pulsed laser. This pressure causes residual compressive stress since it is greater than the yield stress. The enhanced depth of the compressive stress layer with about the same stress magnitude is the main benefit of laser shock processing over traditional shot peening [12].

The limitations of these treatments stem from the challenges posed by focusing on complex form components [13–15]. One of the most promising approaches for achieving homogenous smoothing of intricate objects with internal channels and lattice structures continues to be chemical and electrochemical-based surface treatments [16–18]. It is a pivotal method to remove material that has been polluted by oxygen during processing. In this instance, the resistance to crack initiation and fracture is fundamentally improved by the removal of a hard, brittle top layer. The fundamental benefit of these methods arises from the high mobility of the chemically aggressive solutions, whose smoothing effects can, in certain situations, be more precisely regulated and enhanced if driven by an electric current [19]. As a result, this class of treatments offers the greatest employment opportunity. Several authors exploited chemical polishing to conduct experimental research on the surface finishing of AM components. In a recent study, Tyagi et al. [20] evaluated the chemical polishing of SLM-produced SS316L components and achieved surface roughness reductions of around 92% for both the inner and outer surfaces. Scherillo [21] studied the impact of chemical polishing on AlSi10Mg samples produced by SLM, yielding notable surface quality improvements assessed by a number of surface texture indicators on less complicated geometries. Furthermore, Wysocki et al. [22] demonstrated that chemical machining was efficient in enhancing the surface quality of Ti6Al4V scaffolds produced by EBM while the treatment did not influence the capacity of the surface to integrate bone cells. In a more recent activity, Scherillo et al. [23] proposed a chemical machining approach to treat components produced by Ti6Al4V using EBM. Since HF and HNO_3 are typically used to treat this alloy, the goal of this work was to determine how the ratio of HF to HNO_3 affects the final surface quality, with a particular emphasis on the chemical-physical phenomena that take place throughout the process. The surface quality of Ti6Al4V components produced by EBM has been shown to be drastically improved by chemical machining, and all the solutions used, with varied HF/HNO_3 ratios, have been shown to have smoothing properties. The ultimate quality of a metal surface is improved by the addition of HNO_3, and the decrease of the studied surface parameter increases with increasing HNO_3 concentration. Moreover, it was demonstrated that there are two main phases in the smoothing process. The unfused particles that were left on the surface are removed in the first stage, and the distinctive sharp peaks are selectively dissolved in the second. The solution's transport characteristics have proved to be crucial in producing this outcome; as the concentration of HNO_3 rises, the viscosity increases, and the smoothing effect changes appropriately. Without HNO_3, gaseous H_2 is formed on the metal surface, with the gas bubbles serving as a barrier between the surface and the solution, resulting in a lower material removal rate and worse final quality. A passivation step was further introduced by the injection of HNO_3, and during this stage, ligands were found to be redistributed on the surface, reducing the change in surface composition relative to the bulk alloy. Following what has been done in the previous literature studies, it is therefore important to determine the best approach for reducing surface defects and removing oxides from the surface of titanium components manufactured using different AM technologies and characterized by more complicated geometries. It is also required to identify treatments with a minimal environmental effect that improve aesthetics (in terms of roughness, gloss, etc.) for the creation of design items in titanium alloys.

Materials and methods.

Titanium alloy samples were produced using WAAM technology. In order to improve both the aesthetic appearance and functionality of the samples, a chemical machining and brightening treatment was performed on the samples. Each sample was cut into sections by making two cuts using a metallographic cut machine, using a K-B type grinding wheel. The larger-area specimen was used for the chemical machining treatment, while the remaining two were used for metallographic preparation and as backup reference specimens before performing machining and treatments, as shown in Fig.1.

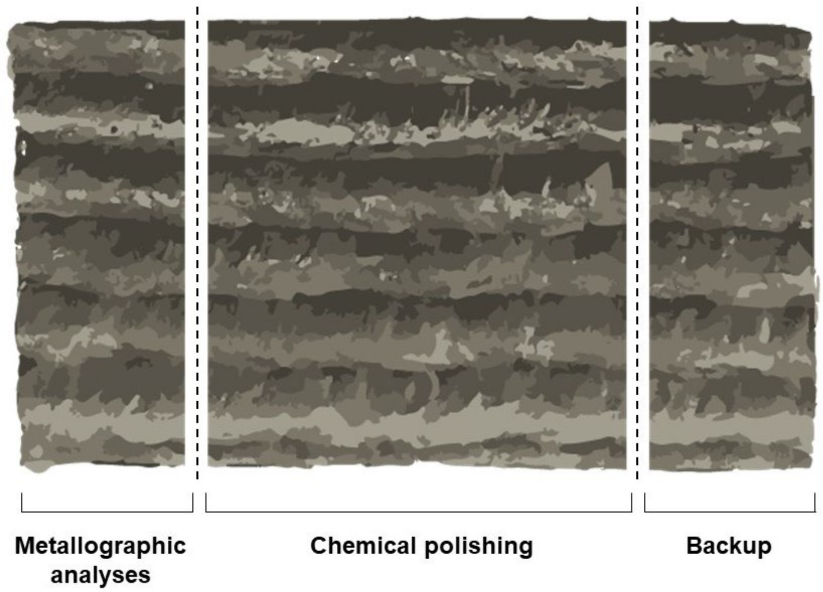


Figure 1. Schematic representation of sample cutting.

The purpose of the analyses performed before the treatment is to evaluate thicknesses to be removed. In fact, when titanium is exposed to an oxidizing atmosphere, in addition to the oxide layer, there is usually an additional layer called the "alpha case," which, due to the presence of oxygen, is harder and therefore more brittle than the bulk material. It is therefore necessary to assess the thickness of the alpha case and remove it completely to prevent the manufactured object from easily surface damage during use. The sample portions used for metallographic analysis were pre-embedded in hot mounting resin; afterwards, the samples were grinded and polished. Subsequently, in order to observe the microstructure, a chemical etching treatment was carried out using a solution made of 1 ml of hydrofluoric acid (48 wt%) and 100 ml of H_2O. The embedded samples were observed employing the optical microscope in order to evaluate the different microstructures present in the border and bulk before treatment and verify the removal of oxides and alpha case after treatment

The chemical treatment was carried out with a solution of hydrofluoric acid (48 wt%) and nitric acid (69 wt%) in a molar ratio of HF/HNO_3 equal to 1 to 3. Treatment times of 30, 60, and 90 minutes were evaluated. The samples were completely immersed in the solution, with the lateral walls shielded using epoxy resin to ensure material removal only on the top and bottom surfaces of the samples. The solution was kept agitated using a magnetic stirrer throughout the treatment. No auxiliary heating or cooling equipment was used, and a sketch of the equipment used for the treatment is shown in Fig.2

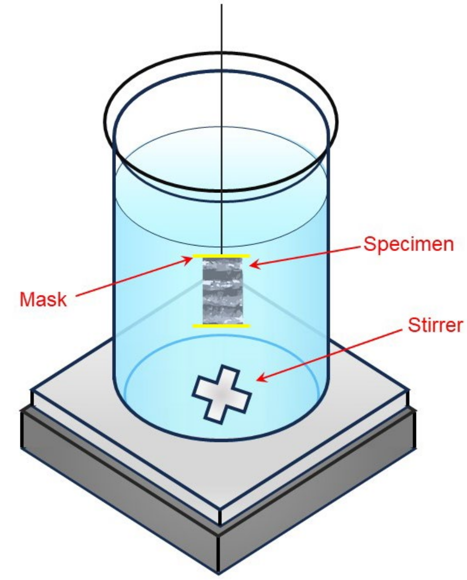


Figure 2. Scheme of the setup for chemical machining treatments.

In order to assess the effectiveness of chemical machining treatment, the samples were analysed, weighed, and measured before and after treatment. The samples were observed under an optical microscope to evaluate their microstructure and measure the thickness of oxide and alpha case. An example of a micrograph of an untreated observed sample is shown in Fig.3.

Result and discussion.
An example of a micrograph of an untreated observed sample is shown in Fig.3.

Figure 3. Optical micrograph of untreated specimen with evident the alpha case.

Fig.3 shows the effect of the different reactivity to the metallographic etching solution between the alpha case and the bulk, as evidenced by the demarcation lines. This allows for a certain determination of the total thickness of the alpha case. It is possible to establish that the thickness of material to be removed, in order to ensure complete removal of the alpha case, is approximately 100 microns.

In order to evaluate the amount of material removed during the treatment, the weights of the samples before and after the treatment were measured. The amount of material removed increases non-linearly with increasing treatment time, as shown in Fig.4, which shows the graphs of the percentage weight loss (Figure 4a) and percentage thickness reduction (Figure 4b) for the different treatment times.

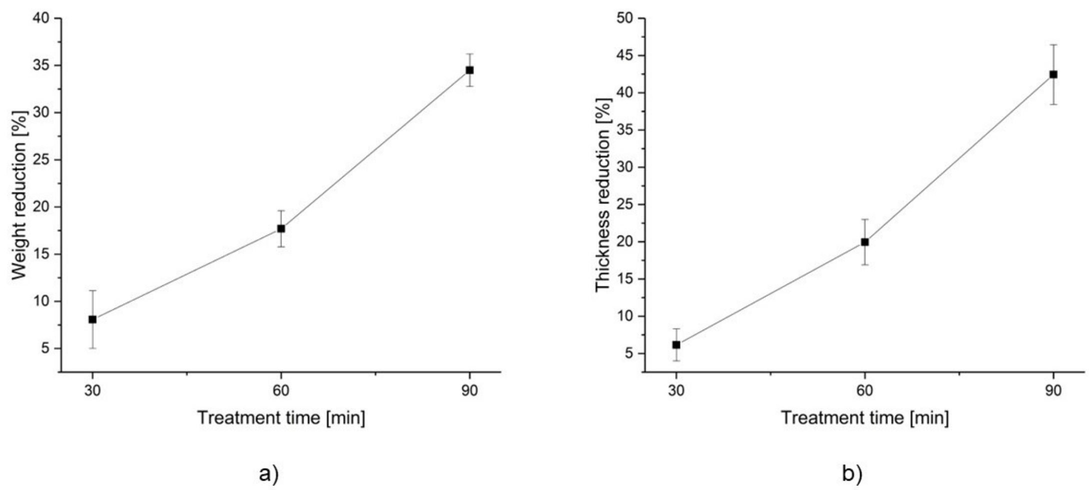


Figure 4. a) Percentage weight loss after 30, 60, and 90 seconds of treatment b) Percentage thickness reduction after 30, 60, and 90 minutes of treatment.

It is evident that the treatment carried out guarantees the removal of a thickness of material from the surface of the samples.

In order to appreciate the variation in brightness of the samples after the chemical treatment and make an initial assessment of the morphological changes of the samples, the images were acquired using a stereomicroscope of the samples before and after treatment. In Fig.5, it is possible to observe the comparisons between samples that underwent machining treatment for 90 minutes.

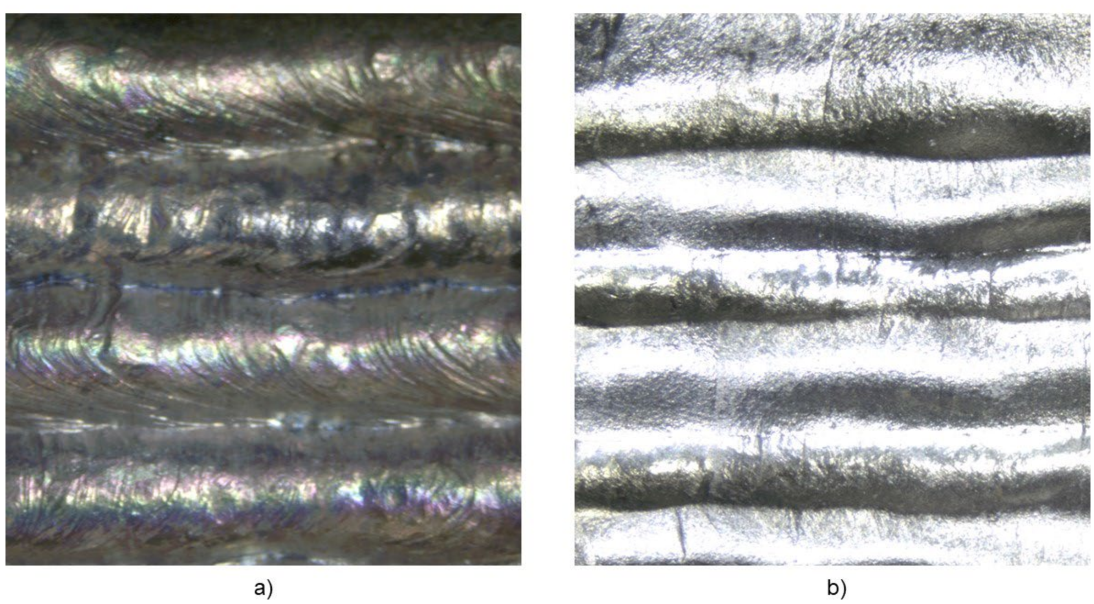
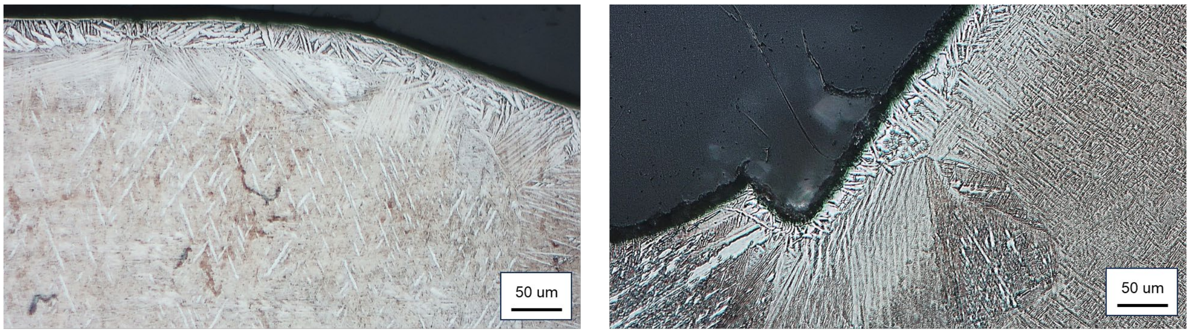


Figure 5. Observation through stereomicroscope of a) Untreated sample b) Sample after a 90-minute chemical machining treatment.

Materials Research Proceedings 41 (2024) 308-315 https://doi.org/10.21741/9781644903131-35

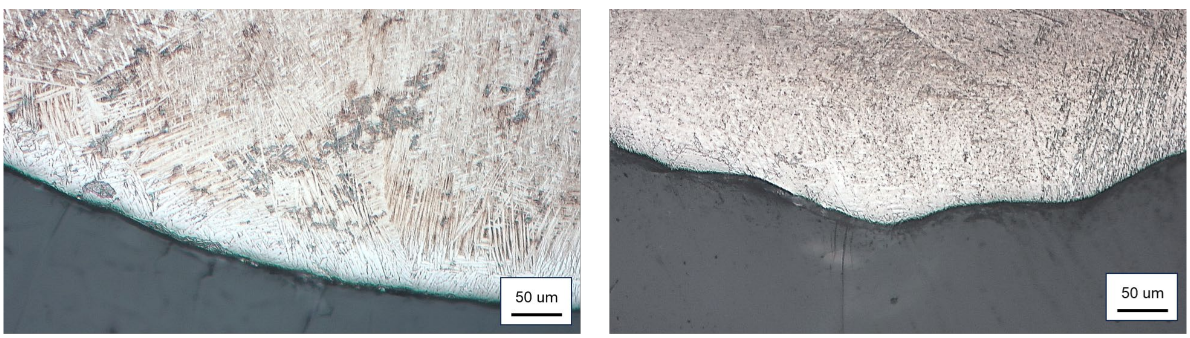
It can be noticed that, as the treatment time increases, the peaks on the surface as well as on the side walls become smoother. After 90 minutes of treatment, the samples appear particularly shiny and smoothed.

The samples were observed under an optical microscope to evaluate the microstructure and the removal of alpha case present at the border. In Fig.6, Fig.7 and Fig.8 it is possible to observe the comparisons between micrographs of samples that underwent machining treatments lasting respectively 30,60 and 90 minutes.



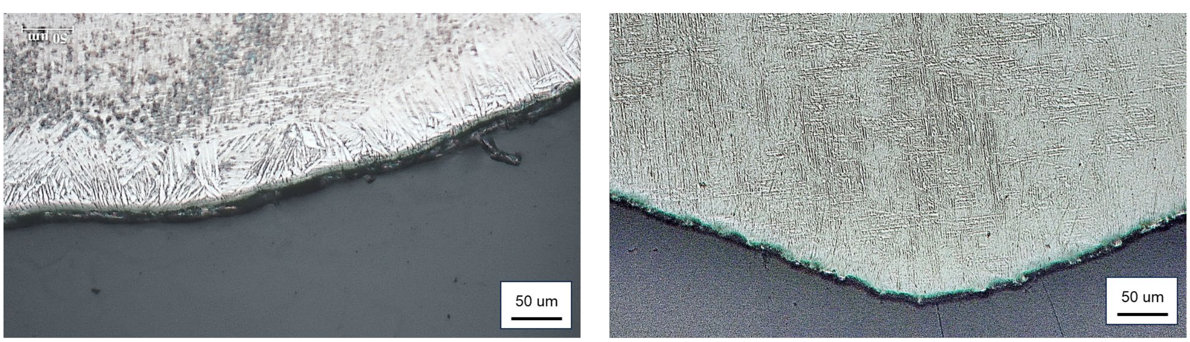
a) b)

Figure 6. Observation under optical microscope of a) Untreated Sample b) Sample after 30 minutes of chemical machining treatment.



a) b)

Figure 7. Observation under optical microscope of a) Untreated Sample b) Sample after 60 minutes of chemical machining treatment.



a) b)

Figure 8. Optical microscope observation of a) Untreated Sample b) Sample after 90 minutes of chemical machining treatment.

It is evident that after 30 minutes of treatment, there is not complete removal of the alpha case. After 60 minutes of treatment, the microstructure at the border appears completely similar to that of the bulk. After 90 minutes of treatment, in addition to the removal of the alpha case, it is also possible to notice morphological modifications of the contour of the samples.

Conclusions

The proposed chemical treatment demonstrates to be effective in removing alpha layer, in particular 90 min are enough to remove 100 µm thickness of alpha layer. Even if more analysis are necessary the surface quality of the sample seems improved, the surface after chemical machining appears very shiny and smooth and this result is of particular interest in many applications.

This study was carried out within the MICS (Made in Italy – Circular and Sustainable) Extended Partnership and received funding from the European Union Next-GenerationEU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR) – MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.3 – D.D. 1551.11-10-2022, PE00000004). This manuscript reflects only the authors' views and opinions, neither the European Union nor the European Commission can be considered responsible for them.

References

[1] M. Pérez, A. García-Collado, D. Carou, G. Medina-Sánchez, R. Dorado-Vicente, On surface quality of engineered parts manufactured by additive manufacturing and postfinishing by machining, in: Addit Manuf, Elsevier, 2021: pp. 369–394. https://doi.org/10.1016/B978-0-12-818411-0.00015-X

[2] A. du Plessis, I. Yadroitsava, I. Yadroitsev, Effects of defects on mechanical properties in metal additive manufacturing: A review focusing on X-ray tomography insights, Mater Des. 187 (2020) 108385. https://doi.org/10.1016/j.matdes.2019.108385

[3] L. Wagner, Mechanical surface treatments on titanium, aluminum and magnesium alloys, Materials Science and Engineering: A. 263 (1999) 210–216. https://doi.org/10.1016/S0921-5093(98)01168-X

[4] Titanium, Springer Berlin Heidelberg, Berlin, Heidelberg, 2007. https://doi.org/10.1007/978-3-540-73036-1

[5] A.I. Nicolas-Silvente, E. Velasco-Ortega, I. Ortiz-Garcia, L. Monsalve-Guil, J. Gil, A. Jimenez-Guerra, Influence of the Titanium Implant Surface Treatment on the Surface Roughness and Chemical Composition, Materials. 13 (2020) 314. https://doi.org/10.3390/ma13020314

[6] O. Unal, A. Cahit Karaoglanli, R. Varol, A. Kobayashi, Microstructure evolution and mechanical behavior of severe shot peened commercially pure titanium, Vacuum. 110 (2014) 202–206. https://doi.org/10.1016/j.vacuum.2014.08.004

[7] P. Peyre, X. Scherpereel, L. Berthe, C. Carboni, R. Fabbro, G. Béranger, C. Lemaitre, Surface modifications induced in 316L steel by laser peening and shot-peening. Influence on pitting corrosion resistance, Materials Science and Engineering: A. 280 (2000) 294–302. https://doi.org/10.1016/S0921-5093(99)00698-X

[8] M. Kobayashi, T. Matsui, Y. Murakami, Mechanism of creation of compressive residual stress by shot peening, Int J Fatigue. 20 (1998) 351–357. https://doi.org/10.1016/S0142-1123(98)00002-4

[9] Y. Yıldıran Avcu, O. Yetik, M. Guney, E. Iakovakis, T. Sınmazçelik, E. Avcu, Surface, Subsurface and Tribological Properties of Ti6Al4V Alloy Shot Peened under Different Parameters, Materials. 13 (2020) 4363. https://doi.org/10.3390/ma13194363

[10] J.Z. Zhou, S. Huang, L.D. Zuo, X.K. Meng, J. Sheng, Q. Tian, Y.H. Han, W.L. Zhu, Effects of laser peening on residual stresses and fatigue crack growth properties of Ti–6Al–4V titanium alloy, Opt Lasers Eng. 52 (2014) 189–194. https://doi.org/10.1016/j.optlaseng.2013.06.011

[11] Q. Hongchao, Experimental investigation of laser peening on Ti17 titanium alloy for rotor blade applications, Appl Surf Sci. 351 (2015) 524–530. https://doi.org/10.1016/j.apsusc.2015.05.098

[12] K.K. Liu, M.R. Hill, The effects of laser peening and shot peening on fretting fatigue in Ti–6Al–4V coupons, Tribol Int. 42 (2009) 1250–1262. https://doi.org/10.1016/j.triboint.2009.04.005

[13] M.K. Thompson, G. Moroni, T. Vaneker, G. Fadel, R.I. Campbell, I. Gibson, A. Bernard, J. Schulz, P. Graf, B. Ahuja, F. Martina, Design for Additive Manufacturing: Trends, opportunities, considerations, and constraints, CIRP Annals. 65 (2016) 737–760. https://doi.org/10.1016/j.cirp.2016.05.004

[14] M. Pérez, D. Carou, E.M. Rubio, R. Teti, Current advances in additive manufacturing, Procedia CIRP. 88 (2020) 439–444. https://doi.org/10.1016/j.procir.2020.05.076

[15] A. Diaz, Surface texture characterization and optimization of metal additive manufacturing-produced components for aerospace applications, in: Additive Manufacturing for the Aerospace Industry, Elsevier, 2019: pp. 341–374. https://doi.org/10.1016/B978-0-12-814062-8.00018-2

[16] E. Maleki, S. Bagherifard, M. Bandini, M. Guagliano, Surface post-treatments for metal additive manufacturing: Progress, challenges, and opportunities, Addit Manuf. 37 (2021) 101619. https://doi.org/10.1016/j.addma.2020.101619

[17] D. Brent, T.A. Saunders, F. Garcia Moreno, P. Tyagi, Taguchi Design of Experiment for the Optimization of Electrochemical Polishing of Metal Additive Manufacturing Components, in: Volume 2: Advanced Manufacturing, American Society of Mechanical Engineers, 2016. https://doi.org/10.1115/IMECE2016-67492

[18] U.S. Kim, J.W. Park, High-Quality Surface Finishing of Industrial Three-Dimensional Metal Additive Manufacturing Using Electrochemical Polishing, International Journal of Precision Engineering and Manufacturing-Green Technology. 6 (2019) 11–21. https://doi.org/10.1007/s40684-019-00019-2

[19] U.S. Kim, J.W. Park, High-Quality Surface Finishing of Industrial Three-Dimensional Metal Additive Manufacturing Using Electrochemical Polishing, International Journal of Precision Engineering and Manufacturing-Green Technology. 6 (2019) 11–21. https://doi.org/10.1007/s40684-019-00019-2

[20] P. Tyagi, T. Goulet, C. Riso, R. Stephenson, N. Chuenprateep, J. Schlitzer, C. Benton, F. Garcia-Moreno, Reducing the roughness of internal surface of an additive manufacturing produced 316 steel component by chempolishing and electropolishing, Addit Manuf. 25 (2019) 32–38. https://doi.org/10.1016/j.addma.2018.11.001

[21] F. Scherillo, Chemical surface finishing of AlSi10Mg components made by additive manufacturing, Manuf Lett. 19 (2019) 5–9. https://doi.org/10.1016/j.mfglet.2018.12.002

[22] B. Wysocki, J. Idaszek, K. Szlązak, K. Strzelczyk, T. Brynk, K. Kurzydłowski, W. Święszkowski, Post Processing and Biological Evaluation of the Titanium Scaffolds for Bone Tissue Engineering, Materials. 9 (2016) 197. https://doi.org/10.3390/ma9030197

[23] F. Scherillo, E. Manco, A. El Hassanin, S. Franchitti, C. Pirozzi, R. Borrelli, Chemical surface finishing of electron beam melted Ti6Al4V using HF-HNO3 solutions, J Manuf Process. 60 (2020) 400–409. https://doi.org/10.1016/j.jmapro.2020.10.033