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SURFACE FINISHING AND ELECTROLESS NICKEL PLATING OF ADDITIVELY MANUFACTURED (AM) METAL COMPONENTS

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ABSTRACT

This study investigates the application of electroless nickel deposition on additively manufactured stainless steel samples. Current additive manufacturing (AM) technologies produce metal components with a rough surface. Rough surfaces generally exhibit fatigue characteristics, increasing the probability of initiating a crack or fracture to the printed part. For this reason, the direct use of as-produced parts in a finished product cannot be actualized, which presents a challenge. Post-processing of the AM parts is therefore required to smoothen the surface. This study analyzes chempolish (CP) and electropolish (EP) surface finishing techniques for post-processing AM stainless steel components CP has a great advantage in creating uniform, smooth surfaces regardless of size or part geometry EP creates an extremely smooth surface, which reduces the surface roughness to the sub-micrometer level.

In this study, we also investigate nickel deposition on EP, CP, and as-built AM components using electroless nickel solutions. Electroless nickel plating is a method of alloy treatment designed to increase manufactured component's hardness and surface resistance to the unrelenting environment. The electroless nickel plating process is more straightforward than its counterpart electroplating.. We use low-phosphorus (2-5% P), medium-phosphorus (6-9% P), and high-phosphorus (10-13% P). These Ni deposition experiments were optimized using the L9 Taguchi design of experiments (TDOE), which compromises the prosperous content in the solution, surface finish, plane of the geometry, and bath temperature. The pre-and post-processed surface of the AM parts was characterized by KEYENCE Digital MicroscopeVHX-7000 and Phenom XL Desktop SEM. The experimental results show that electroless nickel deposition produces uniform Ni coating on the additively manufactured components up to 20 µm per hour. Mechanical properties of as-built and Ni coated AM samples were analyzed by applying a standard 10 N scratch test. Nickel coated AM samples were up to two times scratch resistant compared to the as-built samples. This study suggests electroless nickel plating is a robust viable option for surface hardening and finishing AM components for various applications and operating conditions.

Keywords: additive manufacturing, fatigue, chempolish, electropolish, plating, hardness, Taguchi design of experiments, surface finish.

1. INTRODUCTION

Additive manufacturing (AM) has brought a revolution in the way products are designed and manufactured [1]. Similar to traditionally produced mechanical parts, AM parts are subject to wear, corrosion, fatigue, stress, and shear [2]. Ideally, AM parts should be tough, durable, and corrosion-resistant. However, it is challenging to achieve all the desired mechanical properties by using one material or one process. Some materials may have excellent corrosion resistance but are highly susceptible to stress and load. Other materials could be tough and resistant to deformation but succumb to an acidic or salty environment.

Wear, corrosion and cracks are directly associated with the manufactured part surface [3]. If components have a poor surface finish, the probability of failure is high [4]**.** Post processing techniques like heat treatment, chemical treatment, spray coating, electroplating, and electroless plating are widely used for surface strengthening and corrosion prevention [5][6].

As additive manufacturing transforms design and manufacturing, parts that used to be hard and impossible to manufacture are now possible to manufacture with outstanding precision [7]. Nevertheless, the surface quality of as-produced parts is far from perfect. For this reason, post-processing of additively manufactured parts is inevitable.

It is significantly important for a component to have excellent physical and chemical properties like sufficient surface hardness, low surface roughness, and resistance to a corrosive environment. It is extremely difficult to achieve all the desired physical properties in a single process. Hence, it is necessary to apply multiple processes in a suitable sequence. In the present study, we have applied surface finishing methods to modify the surface quality of AM surfaces so that subsequent coating steps can yield desired results. It is well established that coating can enhance the durability and life of an engineering component [8]. In this paper, we have studied the role of chemical-based surface roughness reduction approaches [9]. To We have chosen chemical-based surface etching methods due to their ability to accomplish surface roughness reductions in hidden areas [10]. Details about our prior work in chemical polishing and electropolishing are published elsewhere [11][12]. Here we also report our method of producing a protective coating on the chemically polished surface. The protective coating can be produced by several methods such as physical vapor deposition [13], electroplating [14][15][16], and electroless plating [17][18]. We have utilized an electroless process for the coating of additively manufactured stainless steel samples. The electroless process is uniquely suitable for producing uniform coatings on complex-shaped components. This paper provides details of experiments for obtaining electroless nickel coatings with high adhesion properties.

2. MATERIALS AND METHODS

The AM pieces were printed at KANSAS CITY NATIONAL SECURITY CAMPUS facility using the EOS® M280 laser sintering-based metal 3D printer. The metal powder used for printing is stainless steel 316 molybdenum alloyed austenitic steel. The alloy comprises 17-19% chromium, 13-15% nickel, 2-3% molybdenum, 6-8% carbon, and balance iron.

2.1 Sample preparation

The sample preparation process involves several stages. First, the samples were sonicated in acetone and isopropyl alcohol (IPA) for 3 min each to remove the impurities and substances on the component's surface such as grease, oil, organic and inorganic components, tarnish, light rust, fingerprints, and oxides. After sonicating with IPA, the samples were cleaned in a bath of sodium hypochlorite (bleach) at 180°F for 2 min to remove residual solvent and oil loosened by pre-cleaning. Following the alkaline cleaning, the samples were electro-cleaned in a heavy-duty ready to use alkaline electrolyte from Krohn industrial inc. The electrolyte bath temperature was kept at 180°F and a 10V potential was applied for one and a half minutes. An oxide layer forms on the samples after electro cleaning. The samples were submerged in HCL for 40 seconds to etch out the oxide layer.

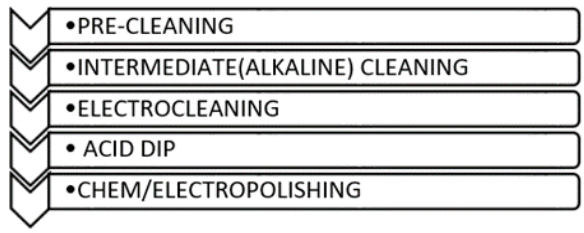


FIGURE 1: Sample Preparation Procedure

2.2 Electropolishing and Chempolishing

CP uses concentrated acidic solution as an electrolyte, however this process is electroless. While the sample is submerged in the bath, the solution anodizes and dissolves the high-stress concentration and crack nucleation regions. Sample surfaces are polished after cleaning process. The chemical bath used for chempolishing comprises 10-30% phosphoric acid, 1-10% hydrochloric acid, 1-10% nitric acid, and 1-10% proprietary surfactants. The optimum bath temperature is 75º C. It is critical to maintain this temperature throughout the process. An increase in temperature could cause an exothermic reaction and contaminate the chemical bath. Agitation plays a significant role in the chempolishing process. It disperses the localized heat generated by the electropolishing process. In our experiments, agitation was done using a 20 mm magnetic stirrer at 200 rpm. After 30 minutes of dissolution, the samples were rinsed in distilled water.

Electropolishing is an electrochemical finishing process that removes a thin layer of material from a metal part. EP is an electrolytic process that uses highly concentrated acidic electrolytes. During the EP process, the sample continually dissolves into the electrolyte. In the case of electropolishing, the electrolyte is composed of 70% phosphoric acid (H_3PO_4) and 30% sulfuric acid (H_2SO_4). The optimum bath temperature for the process is 75º C. Once the solution reaches the desired temperature, the sample (anode) and the lead electrode (cathode) are connected to the power source and a current density of 70 A/dm^2 is applied. The electropolishing is done for 30 minutes, and then an alkaline solution is used to neutralize the sample.

2.3 Electroless Nickel Plating

Generally, there are three types of electroless nickel solutions widely used. The primary determinant of nickel deposition using electroless nickel solutions is the phosphorus content in the solution. In this study, we used low phosphorus

(ONE PLATE 3001), mid phosphorus (ONE PLATE 1001), and high phosphorus (ONE PLATE 2001) electroless nickel plating solution. The chemicals were acquired from plating international Inc. All three baths are very stable, and their pH values range from 5-6. Proper sample cleaning is critical because surface contamination leads to low-quality deposition and prevents good adhesion of the Ni coat. Therefore, the substrate should free of any grease, oil, debris, and oxide layers before the deposition process. These contaminants are removed by cleaning the sample with acetone and isopropyl alcohol. The sample is then ready for the activation process. A few metals, such as zinc, stainless steel, and tungsten, require special pretreatment or activation before plating to ensure maximum adhesion of subsequent plating. To activate our sample, we used Woods nickel strike solution and 5V DC for 30 seconds. The samples are plated immediately after activation.

Electroless plating is a temperature-sensitive process. If the temperatures slightly above the optimal bath temperature, it causes an exothermic reaction, and the chemicals could damage the samples. The optimum temperature for a low and medium phosphorus solution bath is 90^o C, and for high phosphorus solution, the bath temperature is 85^o C. We set the deposition time for all samples to 30 minutes.

2.4 Taguchi Design of Experiment (TDOE)

For this experiment, we used TDOE which gives a set number of different experiments that consist of four parameters and three levels. The first parameter is phosphorus concentration of the electroless nickel solution which is widely exists in three type, low-phosphorous (2-5% P), medium-phosphorus (6-9% P) and high-phosphorus (10-13% P) phosphorus concentration per deposition. The second parameter is the surface finishing associated with reducing surface roughness. Those are EP, CP and as-built surfaces. Third parameter is 3D part coordinate plane. Hence we observe slightly different surface for components produced using selective laser melting process. so we like to investigate how the different plane surface response for the deposition. The final parameter is the temperature of the nickel solution. Generally, there are optimum bath temperature of each solution. However, we like to see how they response to the temperature chance from optimum temperature. So we use temperature five degree Celsius below and above additional to optimum temperature. The TDOE orthogonal arrays reduced the number of the experiments to nine trials. TDOE results in time and resource efficiency, and high quality experiments.

2.5 Testing and Characterization

Scratch test is used for mechanical wear analysis and characterization. Applying well-defined scratches in a reproducible manner is critical when aiming to characterize surfaces' resistance to mechanical wear. We selected a standard 10 N scratch test on the nickel coated samples

We use different characterization and testing on experiment samples. For analyzing the the surface roughness and the depth of the scratch, we use KEYENCE Digital Microscope VHX-7000, and for microstructure and elemental analysis nickel deposited sample, we use Phenom XL Desktop SEM.

Table 1. Taguchi Design of Experiment

DOE	Phosphorus	Surface	Orientation	Temperature
1	Phosphorus content-High	Surface-Electropolish	Plane-XY	Temperature-S+5
2	Phosphorus content-High	Surface-Chempolish	Plane-YZ	Temperature-S
3	Phosphorus content-High	Surface-As build	Plane-XZ	Temperature-S-5
4	Phosphorus content-Mid	Surface-Electropolish	Plane-YZ	Temperature-S-5
5	Phosphorus content-Mid	Surface-Chempolish	Plane-XZ	Temperature-S+5
6	Phosphorus content-Mid	Surface-As build	Plane-XY	Temperature-S
7	Phosphorus content-Low	Surface-Electropolish	Plane-XZ	Temperature-S
8	Phosphorus content-Low	Surface-Chempolish	Plane-XY	Temperature-S-5
9	Phosphorus content-Low	Surface-As build	Plane-YZ	Temperature-S+5

3. RESULTS AND DISCUSSION

The electropolishing process reduces the surface roughness by ~ 92%. This process can remove a large amount of material in a short time. From our roughness measurement, the Ra value reduced from ~ 25 µm to ~ 2 µm. However, the experimental electropolishing process has more dependent variables compared to the chempolish process. These variables include electrolyte concentration, current density, proximity to the electrode, and the type of sample. This could result in non-uniform and inconsistent surface polish.

It is observed chempolishing surface finishing technique resulted in a uniform surface finish on all samples. This is due to the process etches any boundary of the component in contact with same rate and any difficulty. However, the surface was not perfectly smooth. This is due to the low material removal (anodization) rate. The average Ra value for the as-built sample is around 25 µm, whereas after 30 minutes of chempolishing process, the Ra is reduced to ~11 µm. Experimentally, chempolish is easy to apply and only dependent on few variables like concentration and process time.

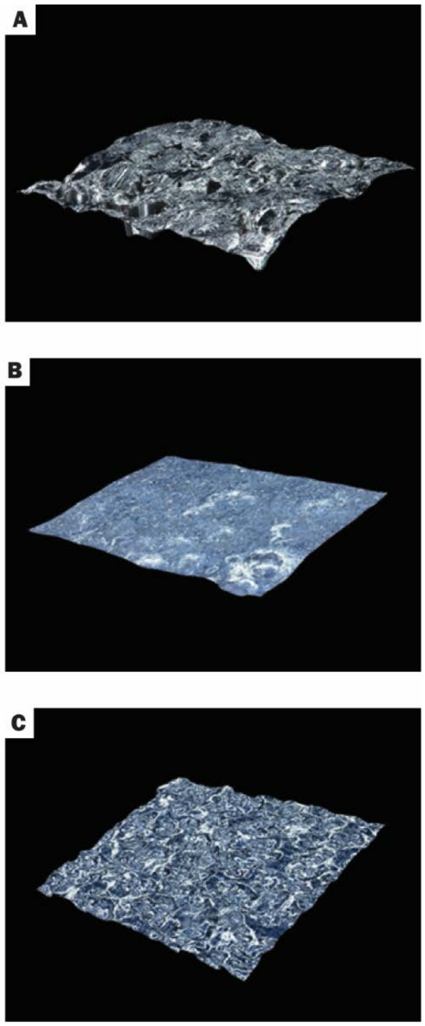


FIGURE 2: (a) As-built AM sample microscopy image (b) Electropolish AM sample microscopy image (b) Chempolish AM sample microscopy image

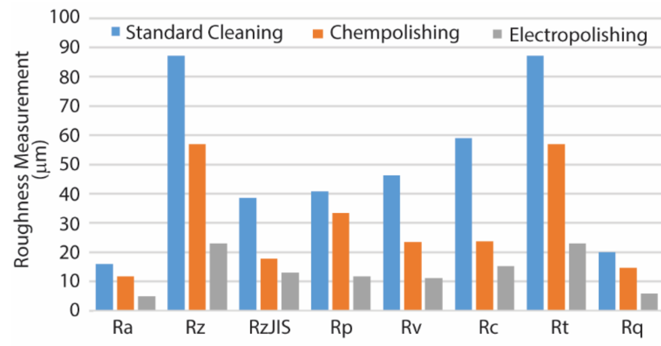
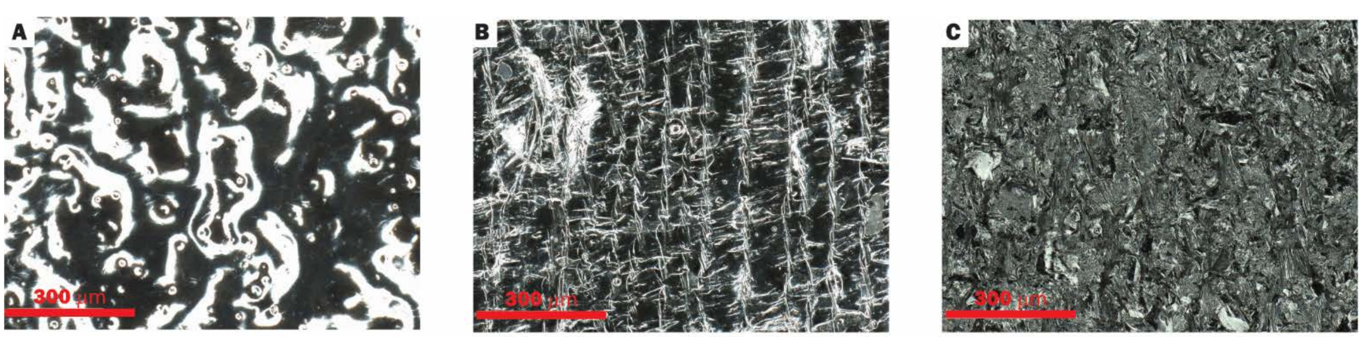


FIGURE 3: Roughness measurement of As-built, electropolish, and chempolish sample

As shown in figure 4(a, b, c) The high phosphorus deposition generally has a low deposition rate. This due to the low nickel content of solution. The rate of the deposition is around 5-7 µm per hour. However, the plating solution is very stable and less responsive to temperature change. Elemental analysis shows the sample contained up to ~ 11% phosphorus concentration per deposition. The coating intends to be amorphous, which implies no grain or phase boundaries to create initiation sites for corrosion. This property makes it very suitable for corrosive and acidic environments. Whereas for CP and as-built sample, a trace amount of deposition is spotted on the substrate.

Figures 4(d, e, f) show medium phosphorus electroless nickel plating on AM stainless steel sample. It observed that medium phosphorus nickel solution has consistent Ni deposition on all three substrates. On average mid phosphorus solution has a plating rate of 15 µm per hour. The elemental analysis on the sample surface shows ~ 8% phosphorus concentration per deposition. This is an excellent alternative plating for balancing corrosion resistance and hard surface.

Figure 4(g, h, i) shows low phosphorus solution on AM stainless steel sample. In contrast, the as-built sample has high adhesion and a high nickel concentration per deposition. The low phosphorus electroless nickel solution has an average deposition rate of 20 µm per hour.



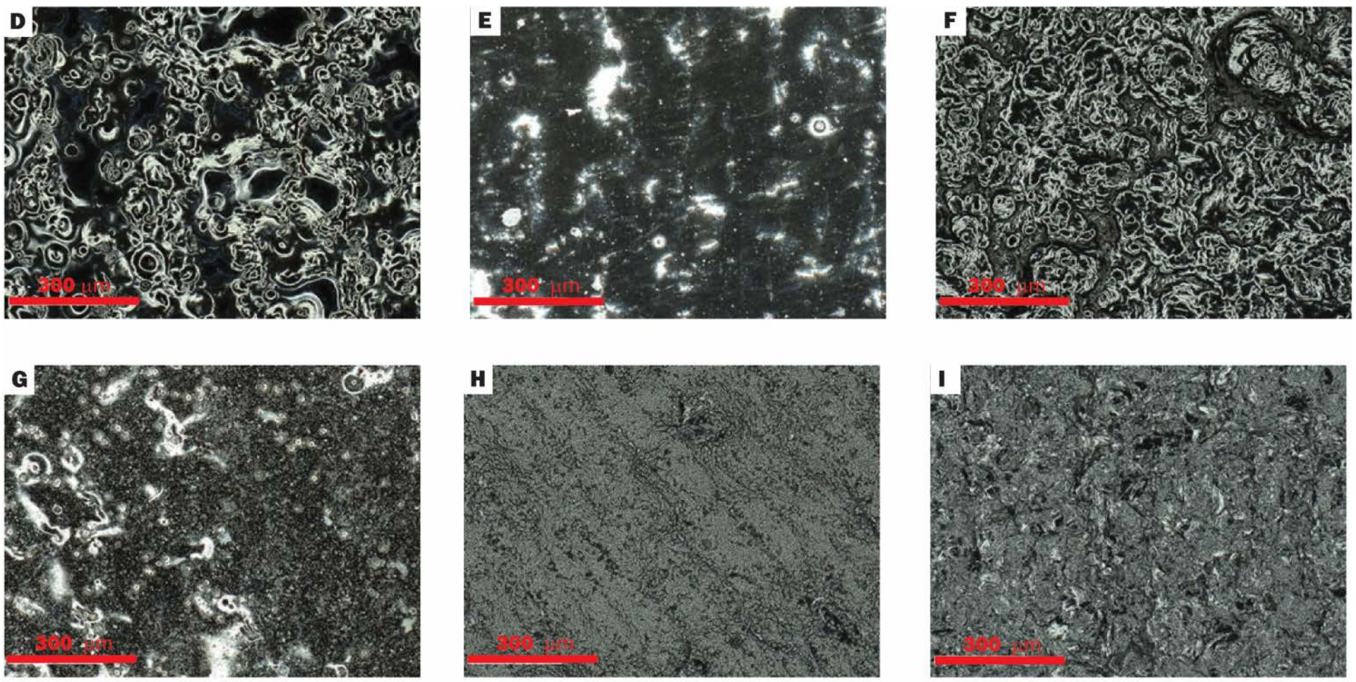


FIGURE 4: Microscopy image for (a) DOE#1 (b) DOE#2 (c) DOE#3 (d) DOE#4 (e) DOE#5 (f) DOE#6 (g) DOE#7 (h) DOE#8 (i) DOE#9

Scratch test Analysis

The standard 10 N scratch test analysis shows significant improvement of resistance for scratch before and after. As shown on the figure 5, DOE #2 and 7 show up to 50% increase in scratch resistance. The DOE #4, 5, 8 and 9 manages to improve the surface hardness from 51% to 86%. The maximum surface hardness improvement is recorded on DOE #6 which is by 128% However, the rest of the experiment didn't show any significant improvement of surface hardness.

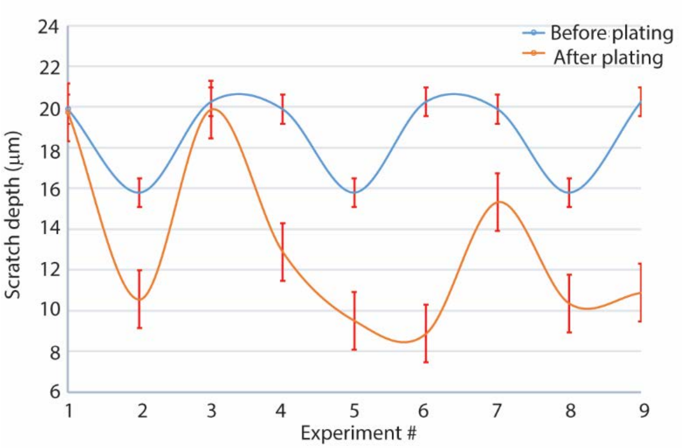


FIGURE 5: Scatter plot for electroless nickel plating before and after

4. CONCLUSION

Electropolishing has excellent surface finishing capability with high material removal rate. However, electropolishing has some limitations with uniformity and repeatability. Chempolishing is one of the best alternatives for surface finishing due to its uniform material removal rate and the potential of smoothening internal and external surfaces.

Electroless nickel deposition has superior plating potential on additively manufactures stainless samples. Nickel offers excellent wear resistance. We observe nickel plated samples up to two times scratch resistant through the scratch testing process than as produced samples. The high phosphorus electroless nickel solution gives extra corrosion resistance. We conclude that the geometry of the printed part highly influences the surface finishing process.

Finally, applying successive surface finishing techniques results in a shiny and smooth surface with excellent surface hardness and corrosion resistance workpiece.

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REFERENCES

[1] E. Govekar, A. Jeromen, A. Kuznetsov, M. Kotar, and M. Kondo, "Annular laser beam based direct metal deposition," *Procedia CIRP*, vol. 74, pp. 222–227, 2018, doi: 10.1016/j.procir.2018.08.099.

[2] H. R. Lashgari, Y. Xue, C. Onggowarsito, C. Kong, and S. Li, "Microstructure, Tribological Properties and Corrosion Behaviour of Additively Manufactured 17-4PH Stainless Steel: Effects of Scanning Pattern, Build Orientation, and Single vs. Double scan," *Mater. Today Commun.*, vol. 25, no. August, 2020, doi: 10.1016/j.mtcomm.2020.101535.

[3] S. G. Croll, "Surface roughness profile and its effect on coating adhesion and corrosion protection: A review," *Prog. Org. Coatings*, vol. 148, no. April, 2020, doi: 10.1016/j.porgcoat.2020.105847.

[4] P. Tyagi *et al.*, "Reducing the roughness of internal surface of an additive manufacturing produced 316 steel component by chempolishing and electropolishing," *Addit. Manuf.*, vol. 25, pp. 32–38, 2019.

[5] "8 Metal Surface Finishing Treatment Processes | Keller Technology." https://www.kellertechnology.com/blog/8-common-types-of-surface-treatments-for-metal-parts/ (accessed Jun. 30, 2021).

[6] "Metal Finishing–What Types of Finishes Are There?" https://www.thomasnet.com/articles/custom-manufacturing-fabricating/types-metal-finishing/ (accessed Jun. 30, 2021).

[7] G. Bansal, D. B. Singh, H. S. Virk, A. Devrani, and A. Bhandari, "Microstructural characterization, applications and process study of various additive manufacturing process: A review," *Mater. Today Proc.*, vol. 26, pp. 833–837, 2019, doi: 10.1016/j.matpr.2020.01.048.

[8] "Benefits of Industrial Plating and Coating - Industry Today %." https://industrytoday.com/benefits-of-industrial-plating-and-coating/ (accessed Jul. 01, 2021).

[9] T. G. C. R. R. S. N. C. J. S. C. B. F. G.-M. P. Tyagi∗, "Reducig the roughess of internal surface of an additive manufacturing produced steel component by chempolishing and electropolishing," *ASME*, 2018.

[10] V. Urlea and V. Brailovski, "Electropolishing and electropolishing-related allowances for powder bed selectively laser-melted Ti-6Al-4V alloy components," *J. Mater. Process. Technol.*, vol. 242, pp. 1–11, 2017, doi: 10.1016/j.jmatprotec.2016.11.014.

[11] "What is Electropolishing? How Does Electropolishing Work?" https://www.besttechnologyinc.com/electropolishing-equipment/how-does-electropolishing-work/ (accessed Jun. 22, 2021).

[12] J. Dillard, A. Grizzle, W. Demisse, P. Tyagi, L. Rice, and C. Benton, "Effect of Altering the Sequence of Chempolishing and Electropolishing on Surface Properties of Additively Manufactured (AM) 316 Steel Components." Nov. 16, 2020, doi: 10.1115/IMECE2020-23878.

[13] P. Lassègue *et al.*, "Laser powder bed fusion (L-PBF) of Cu and CuCrZr parts: Influence of an absorptive physical vapor deposition (PVD) coating on the printing process," *Addit. Manuf.*, vol. 39, p. 101888, 2021, doi: https://doi.org/10.1016/j.addma.2021.101888.

[14] W. Han, X. Qian, H. Ma, X. Wang, and Y. Zhang, "Effect of nickel electroplating followed by a further copper electroplating on the micro-structure and mechanical properties of high modulus carbon fibers," *Mater. Today Commun.*, vol. 27, p. 102345, 2021, doi: https://doi.org/10.1016/j.mtcomm.2021.102345.

[15] J.-C. Hung, H.-P. Tsui, P.-C. Chen, and P.-J. Yang, "Using a nickel electroplating deposition for strengthening microelectrochemical machining electrode insulation," *J. Manuf. Process.*, vol. 67, pp. 77–90, 2021, doi: https://doi.org/10.1016/j.jmapro.2021.04.056.

[16] T. Yamamoto *et al.*, "Effects of current density on mechanical properties of electroplated nickel with high speed sulfamate bath," *Microelectron. Eng.*, vol. 213, pp. 18–23, 2019, doi: https://doi.org/10.1016/j.mee.2019.04.012.

[17] W. Sha, X. Wu, and K. G. Keong, "10 - Crystallisation of nickel–phosphorus (Ni–P) deposits with high phosphorus content," in *Woodhead Publishing Series in Metals and Surface Engineering*, W. Sha, X. Wu, and K. G. B. T.-E. C. and N. P. Keong, Eds. Woodhead Publishing, 2011, pp. 141–162.

[18] D. G. Agredo Diaz *et al.*, "Effect of a Ni-P coating on the corrosion resistance of an additive manufacturing carbon steel immersed in a 0.1 M NaCl solution," *Mater. Lett.*, vol. 275, p. 128159, 2020, doi: 10.1016/j.matlet.2020.128159.