Proceedings of the ASME 2023
International Mechanical Engineering Congress and Exposition
IMECE2023
October 29-November 2, 2023, New Orleans, Louisiana

IMECE2023-114338

ELECTROPOLISHING (EP), CHEM POLISHING (CP), AND AS-BUILT ADDITIVELY MANUFACTURED METAL COMPONENTS FOR ELECTROLESS NICKEL PLATING RESEARCH

Pablo Sanchez
Mechanical Engineering,
University of the District of Columbia
Washington, DC, 20008, USA

Lucas Rice
Mechanical Engineering,
Kansas City National Security Campus
Kansas City, MO 64147, USA

Pawan Tyagi
Mechanical Engineering,
University of the District of Columbia
Washington, DC, 20008, USA

ABSTRACT

The current study investigates electroless nickel plating and surface finishing techniques such as ChemPolishing (CP) and ElectroPolishing (EP) for postprocessing on additively manufactured stainless-steel samples. Existing additive manufacturing (AM) technologies generate metal components with a rough surface that typically exhibit fatigue characteristics, resulting in component failure and undesirable friction coefficients on the printed part. Small cracks formed in rough surfaces at high surface roughness regions act as a stress raiser or crack nucleation site. As a result, the direct use of as-produced parts is limited, and smoothening the Surface presents a challenge. Previous research has shown that CP ChemPolishing has a significant advantage in producing uniform, smooth surfaces regardless of size or part geometry. EP Electropolishing has a high material removal rate and an excellent surface finishing capability. Electropolishing, on the other hand, has some limitations in terms of uniformity and repeatability. On additively manufactured stainless-steel samples, electroless nickel deposition has a higher plating potential. Nickel has excellent wear resistance, and nickel-plated samples are more robust as scratch resistant than not plated samples when tested for scratch resistance. This research uses medium-phosphorus (6-9% P) and high-phosphorus (10-13% P). The L9 Taguchi design of experiments (DOE) was used to optimize the electroless nickel deposition experiments. The mechanical properties of as-built and nickel-coated additive manufacturing (AM) samples were investigated using a standard 5 N scratch test and the adhesion test ASTM B-733 thermal shock method. The KEYENCE Digital Microscope VHX-7000 was used to examine the pre- and post-processed surfaces of the AM parts. The complete scratch and Design of Experiment (DOE) analysis was performed using the Qualitek-4 software. This work is in progress concerning testing the optimum conditions, completing measurements, and analyzing the results.

Keywords: Taguchi design of experiments, additive manufacturing, chem polishing, electropolishing, electroless nickel plating, hardness, crack nucleation.

1. INTRODUCTION

Additive manufacturing (AM) has changed the way products are created and manufactured [1]. Wear, corrosion, fatigue, stress, and shear are all factors that affect AM parts, just as they do traditional manufactured mechanical part [2]. In general, AM components should be durable, long-lasting, and corrosion-resistant. However, achieving all of the desired mechanical properties and qualities with a single material or technique may be complex and difficult as well. Some of these materials are quite corrosion-resistant, but they are extremely vulnerable to stress and load on the negative side. Some materials are tough and can withstand strain without being permanently damaged, but they degrade in an acidic or corrosive environment.

In general, engineering defiance occurs due to the surface of the manufactured part [3]. If components have poor surface quality, there is a high risk of failure [4]. Engineers have been attempting to devise a variety of solutions and post-processing approaches to address these issues since the dawn of engineering. In this regard, heat and chemical treatment, spray coating, electroplating, and electrodeposition or electroless plating are commonly used available techniques.

Parts that were previously difficult or impossible to create are now easily manufactured with exceptional accuracy thanks to additive manufacturing, which has revolutionized the design and production process [5]. However, there is still room for improvement in the surface quality of manufactured parts. As a result, post-processing of additively manufactured parts is critical.

This article investigates various surface finishing techniques, both individually and collectively. Surface finishing techniques used include electropolishing, ChemPolishing, and combining those finishing procedures with electroless nickel plating. The experiments will be aided by the use of a variety of surface finishing to determine the best physical attribute.

The electropolishing (EP) procedure tends to remove a thin coating of material from a metal part [6]. The EP process uses highly concentrated acidic electrolytes and DC electric current in a closed circuit. During operation, the power supply causes electrons to flow from the anode (sample) to the cathode (electrode), and the sample dissolves into the electrolyte on an ongoing basis. In the absence of a power supply, Chempolishing employs a highly acidic solution as an electrolyte. As the sample is immersed in the bath, the solution anodizes and dissolves the high-stress concentration and crack nucleation zone [7].

To implement electroless nickel plating, Brenner and Riddel were the first to try [8]. Afterward, it has been widely employed in a variety of sectors, including the aircraft industry, automobiles, and textiles [9]. Electroless Nickel-Phosphorus (Ni-P) plating's commercial success is mainly due to the process's unique qualities and the specific attributes of (Ni-P) coatings. There are several potential plating surfaces for different circumstances and applications by simply changing different combinations of nickel and phosphorous [10].

2. MATERIALS AND METHODS

The stainless steel 316 molybdenum alloyed austenitic steel was used as the printing sample material. This alloy has a chromium content of 17-19%, a nickel content of 13-15%, a molybdenum content of 2-3%, a carbon content of 6-8%, and a high iron content of 55-57%. The Kansas City National Security Campus is where the sample parts are made. The samples were made using the EOS® M280 laser sintering-based additive manufacturing equipment. All materials and methods that have been used in the work must be stated clearly.

2.1 Sample Preparation

The recommendation in the Standard Guide for Cleaning Metals Designation B322 99 (Reapproved 2014.) was followed for sample preparation prior to Electroplating. This action was realized to remove contaminants or chemicals on the surface of the component, such as grease, oil, organic and inorganic components, fingerprints, and oxides since the sample must be thoroughly cleaned. The sample preparation procedure was broken down into several sequential steps.

The first step, known as pre-cleaning, is intended to remove a significant amount of oil, particularly buffing compound, or grease deposits. At this point, samples were soaked in acetone for 3 minutes. The grease, oil, ink, permanent marker, adhesive, and paint were all dissolved by the acetone. The samples are then rinsed with distilled water and dried with an air-drying apparatus. Following that, the samples are cleaned for 3 minutes in the digital ultrasonic cleaner apparatus with 99% isopropyl alcohol (IPA). The residual contaminants on the sample are dissolved during this process. The cleaning procedures then proceed to intermediate alkaline cleaning and electro cleaning. For intermediate-alkaline cleaning, a solution bath of sodium hypochlorite (bleach) at 180°F for approximately 2 minutes is used. It removes any remaining solvent or oil that may have been softened or conditioned during the pre-cleaning process. After that, the sample is washed and dried. The following stage is electro-cleaning (EC). This electrochemical cleaning technique employs an alkaline electrolyte and direct current (DC). Krohn Industrial Inc's heavy-duty ready-to-use solution was used in the tests. The bath temperature was kept at 180°F and the bias voltage was held at 10V for one and a half minutes. Following electrocleaning, an oxide layer forms on the sample. A quick acid dip is required to remove the oxide layer and neutralize the sample. The oxide layer was etched away by immersing the sample in HCL for 40 seconds. The sample cleaning procedure involved precleaning with organic solutions, intermediate cleaning with alkaline solutions, electrocleaning, acid cleaning, and ChemPolishing or electropolishing. The electropolishing, chemical polishing, and electroless nickel plating process has been discussed in our prior publication [2022 ASME paper.]

2.2 Taguchi Design of Experiment (TDOE) Application

Taguchi Design of Experiment (TDOE) was applied for this project, which provides a defined number of possible experiments with four parameters and three levels, as represented in Table 1. The chosen parameters were as follows: the bath's phosphorus content, Nickel strike time of bath exposure, pre-surface polish, and the 3D coordinate plane.

Table 1: TDOE with levels 1, 2, 3 for various impact factors.

Factors	Level 1	Level 2	Level 3
Nickel Strike Time	30	45	60
Surface Preparation	Chem-Po	Electro-Po	Asproduced
Orientation	XY	YZ	XZ
P Level 1	High	Mid	Mid
P Level 2	High	Mid	High

The design of experiments and trial conditions are illustrated in Table 2. As shown in Table 2, the L9 TDOE orthogonal array cuts the number of trials to nine. Time and resource efficiency, as well as high-quality trials, are the outcomes of TDOE.

Table 2: Design of experiments and trial conditions.

DOE	Randomly selected order (X)	Factors	Levels
1	Nickel Strike Time	30	1
	Surface Preparation (9)	Chem-Polishing	1
	Orientation	XY	1
	P Level (Nickel Solution)	Mid	1
2	Nickel Strike Time	30	1
	Surface Preparation (3)	Elec-Polishing	2
	Orientation	YZ	2
	P Level (Nickel Solution)	High	2
3	Nickel Strike Time	30	1
	Surface Preparation (8)	As-Built	3
	Orientation	XZ	3
	P Level (Nickel Solution)	Mid	1
4	Nickel Strike Time	45	2
	Surface Preparation (7)	Chem-Polishing	1
	Orientation	YZ	2
	P Level (Nickel Solution)	Mid	1
5	Nickel Strike Time	45	2
	Surface Preparation (5)	Elec-Polishing	2
	Orientation	XZ	3
	P Level (Nickel Solution)	Mid	1
6	Nickel Strike Time	45	2
	Surface Preparation (4)	As-Built	3
	Orientation	XY	1
	P Level (Nickel Solution)	High	2
7	Nickel Strike Time	60	3
	Surface Preparation (2)	Chem-Polishing	1
	Orientation	XZ	3
	P Level (Nickel Solution)	High	2
8	Nickel Strike Time	60	3
	Surface Preparation (6)	Elec-Polishing	2
	Orientation	XY	1
	P Level (Nickel Solution)	Mid	1
9	Nickel Strike Time	60	3
	Surface Preparation (1)	As-Built	3
	Orientation	YZ	2
	P Level (Nickel Solution)	Mid	1

3. RESULTS AND DISCUSSION

3.1 Scratch Test Analysis

It is critical to apply well-defined scratches in a consistent manner when testing a surface's resistance to mechanical wear or coating. The scratch test is used to characterize and analyze mechanical wear or coatings. However, the results are affected by a number of factors, including the thickness of the coating, the mechanical properties of the substrate, the strength of the interfacial bond, and test conditions such as the speed at which the scratch occurs, the load it is subjected to, and the radius of the indenter tip. The nickel-plated samples were subjected to a standard 5 N scratch test with the TABER 5900 reciprocating abraser. Figure 1 exhibits an illustration of a scratch performed on an AM sample.

FIGURE 1: ILLUSTRATION OF A SCRATCH PERFORMED ON AN AM SAMPLE.

Figure 2 reveals a scatter plot for the scratch test executed before and after electroless nickel deposition.

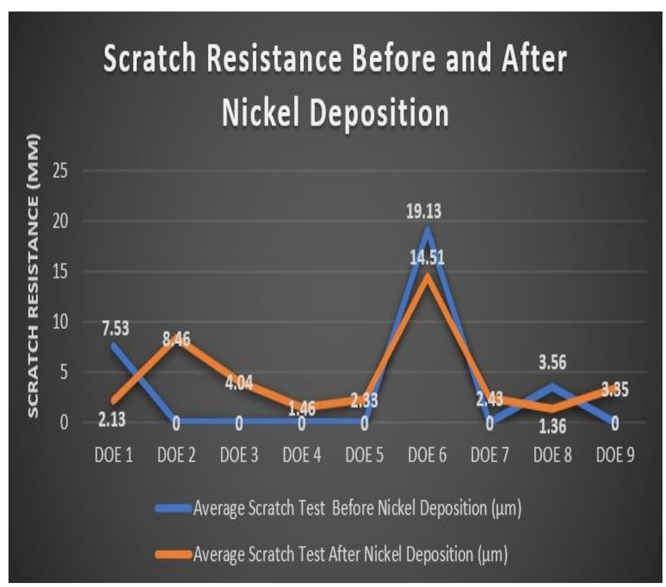

FIGURE 2: SCATTER PLOT FOR THE SCRATCH TEST EXECUTED BEFORE AND AFTER ELECTROLESS NICKEL DEPOSITION.

It is clear that the scratch depth decreases after nickel deposition. Consider three cases; DOE 1 - surface preparation Chem-Polishing, DOE 6 - surface preparation As-Built , and DOE 8 - surface preparation Elec-Polishing. In DOE 1, the before and after nickel deposition scratch depths results were 7.53 and 2.13 microns, DOE 6 19.13 and 14.51 microns, and DOE 8 3.56 and 1.36 microns, respectively. These results corroborate the effectiveness of electroless nickel plating as an alloy treatment method designed to increase AM components' hardness and surface resistance.

3.2 Taguchi Design of Experiment (TDOE) analysis, and optimization

Taguchi Design of Experiment (DOE) is a statistical methodology used to optimize the performance of a system or process using the fewest number of experiments possible. Taguchi DOE is divided into three stages: design, analysis, and optimization. A set of experimental conditions is determined during the design stage based on the system or process's input factors (also known as control factors) and output responses (also known as quality characteristics). An orthogonal array, which is a table containing a set of level combinations for each input factor, is used to create the design. The performance of the system or process is measured under each set of experimental conditions during the analysis stage. The collected data is then analyzed statistically to identify the most significant input factors and their levels that influence the output responses. The optimal combination of input factor levels is determined in the optimization stage to achieve the desired output responses with the least variation. This is accomplished by employing a signal-to-noise (S/N) ratio, which is a measure of the quality of output responses in relation to data variability.

Figure 3 indicates the Qualitek-4 Ra roughness surface result analysis.

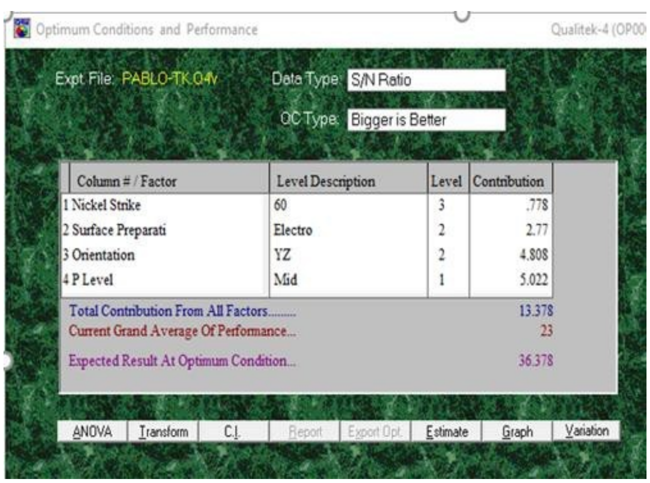

FIGURE 3: QUALITEK-4 RA ROUGHNESS SURFACE RESULT.

It determined that the optimum condition for roughness improvements will be reached at Nickel Strike 60 seconds-level 3, Surface Preparation Chem Polishing-level 1, Orientation YZ-level 2, Phosphorus level Medium-level 1. Likewise, Figure 4 illustrates the optimal condition will improve S/N ratio from -20. 733 in the current condition to -11.081 improved condition and the standard deviation from 10.762 current condition to 3.542 improved condition with a target of 12.279.

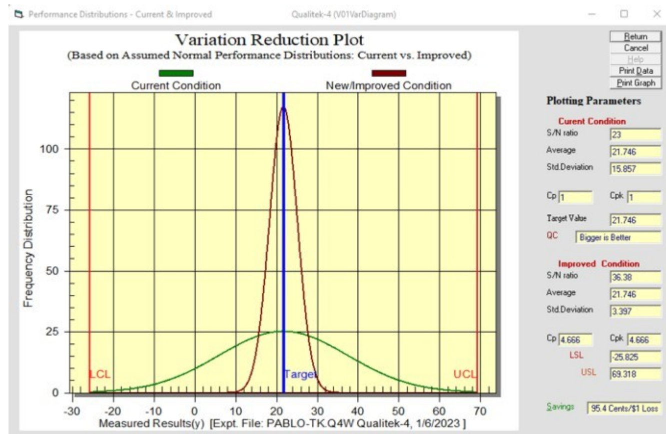

FIGURE 4: QUALITEK-4 RA ROUGHNESS SURFACE OPTIMAL CONDITION RESULT.

Correspondingly, for the Qualitek-4 nickel deposition layer thickness result analysis, Figure 5 display that the optimum condition will be attained at Nickel Strike 60 seconds-level 3, Surface Preparation ElectroPolishing-level 1, Orientation YZ-level 2, Phosphorus level Medium-level 1. Similarly, Figure 6 shows the optimal condition will improve S/N ratio from 23 in the current condition to 36.38 improved condition and the standard deviation from 15.857 current condition to 3.397 improved condition with a target of 21.746.

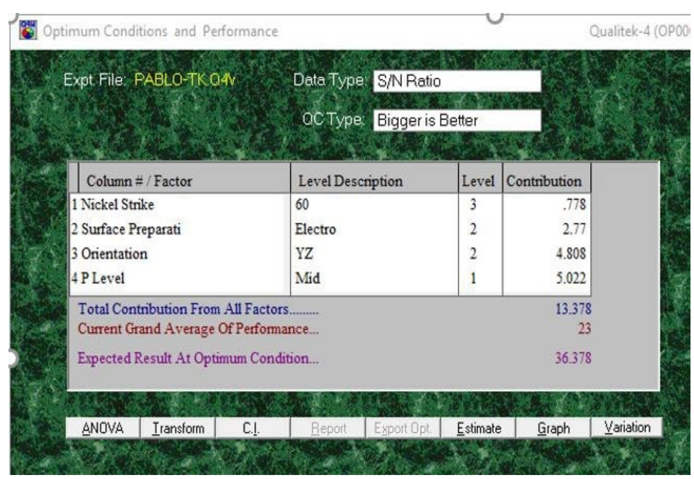

FIGURE 5: QUALITEK-4 NICKEL DEPOSITION LAYER THICKNESS.

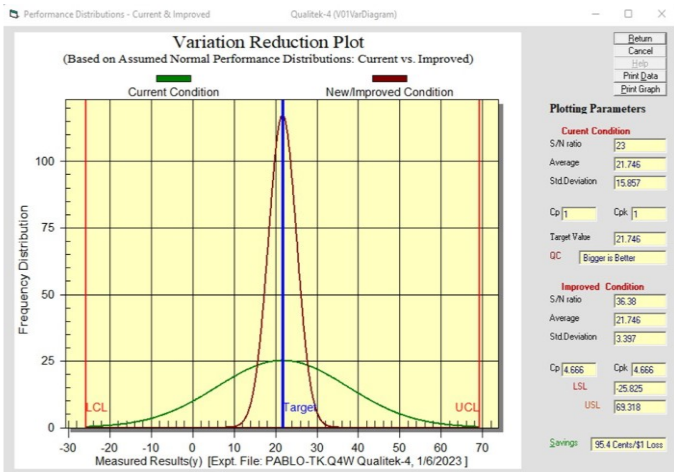

FIGURE 6: QUALITEK-4 NICKEL DEPOSITION LAYER THICKNESS OPTIMAL CONDITION RESULT.

4. CONCLUSION

Chempolishing is one of the most effective surface finishing options owing to its consistent material removal rate and ability to smoothen interior and exterior surfaces. Electropolishing provides a high material removal rate and strong surface finishing capabilities. On the other hand, Electropolishing has some limits in terms of consistency and reproducibility. The Electropolishing method reduces surface roughness by 63.8 %. The Arithmetic Average Roughness or the absolute average relative to the base length Ra value was decreased from 16.81µm to 6.09 µm as done in the roughness measurements. On the other hand, the absolute average Ra value for the as-built sample is roughly 16.81µm, which decreased after applying the ChemPolishing process to 10.52 µm. It represents a 37.41 % decrease in surface roughness.

The Qualitek-4 Ra roughness surface result shows that the predictive equation for performance at the optimum condition and any other possible condition will be achieved at Nickel strike time 60 second, Surface preparation Chem polishing, Orientation YZ, P level Mid. The most significant factor and interaction influences were surface preparation, followed by nickel strike time, orientation, and P level.

The Qualitek-4 nickel deposition layer thickness analysis results shows that the predictive equation for performance at the optimum condition and any other possible condition will be achieved at Nickel strike time 60 second, Surface preparation Electro polishing, Orientation YZ, P level Mid. The most significant factor and interaction influences were orientation, followed by P level, surface preparation, and nickel strike time.

The plating rate of mid phosphorus solution is 15 µm per hour on average. The elemental analysis on the sample surface reveals a phosphorus content of 8.5 % per deposition. This is an excellent plating choice for balancing corrosion resistance and hardness.

The adhesion test ASTM B-733 thermal shock method utilized in this research demonstrated the Electroless Nickel Plating process performed achieved excellent adhesion of the layer since the coating was examined for blistering or other evidence of poor adhesion and did not find any evidence of it.

Thus, the electroless nickel deposition provides more significant plating potential on additively manufactured stainless samples. Nickel has high wear resistance. The electroless nickel solution with a high phosphorus content provides further corrosion resistance. It was found that the printed part's shape has a significant impact on the surface finishing process.

ACKNOWLEDGEMENTS

The partial financial support is gratefully acknowledged from National Science Foundation-CREST Award (Contract # HRD- 1914751), Department of Energy/ National Nuclear Security Agency (DE-FOA-0003945). The Department of Energy's Kansas City National Security Campus is the primary

funder of this initiative. The Kansas City National Security Campus is managed and operated by Honeywell Federal Manufacturing & Technologies, LLC under contract number DE-NA0002839 with the Department of Energy.

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, 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., vol. 25, pp. 32–38, 2019.

[5] G. Bansal, D.B. Singh, H.S. Virk, A. Devrani, 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.

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

[7] 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.

[8] "The Evolution of Electroless Nickel Plating | CT." https://coatingtechnologiesllc.com/2017/03/the-evolution-of-electroless-nickel-plating/ (accessed Jun. 22, 2021).

[9] Z. Huang, T. T. Nguyen, Y. Zhou, and G. Qi, "A low-temperature electroless nickel plating chemistry," Surf. Coatings Technol., vol. 372, no. April, pp. 160–165, 2019, doi: 10.1016/j.surfcoat.2019.05.019.

[10] W. Sha, X. Wu, and K. G. Keong, "10 - Crystallization 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.