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ENHANCING SURFACE FINISHING OF ADDITIVELY MANUFACTURED METAL COMPONENTS THROUGH ELECTROLESS NICKEL PLATING AND MACHINE LEARNING-ASSISTED INSTANCE SEGMENTATION

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ABSTRACT

This research investigates the feasibility of electroless nickel deposition on additively manufactured stainless steel samples. The prevalent additive manufacturing techniques for metal components generate a surface with rough surface, which can result in a higher likelihood of fatigue and the initiation of cracks or fractures in the printed part. As a result, using as-built components as final product is impractical, which requires post-processing to create a smoother surface. This study assesses chempolishing (CP) and Electropolish (EP) techniques for post-processing additively manufactured stainless steel components. CP is a purely chemical process that involves continuous anodization of the sample, resulting in oxidation-reduction. Conversely, EP is an electrochemical process that necessitates an electric current to facilitate polishing. EP produces an exceptionally smooth surface that reduces surface roughness to a sub-micrometer level. We observed that EP and CP techniques reduced the surface roughness's arithmetical mean height (Ra) by up to 4 µm and 10 µm, respectively.

In this study, we also investigate the application of electroless nickel deposition on AM components using different surface finishing techniques, including EP, CP, and as-built components. Electroless nickel plating aims to enhance the surface hardness and resistance of manufactured components to withstand harsh environmental conditions, we used low-phosphorus (2-5% P), medium-phosphorus (6-9% P), and high-phosphorus (10-13% P) nickel solutions. we utilized the L9 Taguchi design of experiments (TDOE) to optimize these Ni deposition experiments, which consider solution content, surface finish, geometry plane, and bath temperature. The pre-and post-processed surfaces of the AM parts were analyzed using the KEYENCE Digital Microscope VHX-7000 and Phenom XL Desktop SEM. We apply a machine learning-based instance segmentation technique to improve the identification of nickel deposition and surface topology of microscopic images. Our experiments show that electroless nickel deposition produces uniform Ni coating on the additively manufactured components at up to 20 µm per hour. Mechanical properties of as-built and Ni-coated AM samples were evaluated using a standard 10 N scratch test. It was found that the nickel-coated AM samples were up to two times more scratch-resistant than the as-built samples. Based on our findings, we conclude that electroless nickel plating is a robust and viable option for surface hardening and finishing AM components for various applications and operating conditions.

Keywords: Additive manufacturing, plating, wear, AI, image processing

1. INTRODUCTION

Additive manufacturing (AM) has significantly transformed product design and development [1]. Although AM parts has increased the efficiency of the parts, they are still prone to wear, corrosion, fatigue [2], stress [3], and shear [4]. It is highly desirable that AM parts possess attributes such as toughness, durability, and corrosion resistance. Nevertheless, accomplishing all the targeted mechanical properties through a single material or process is impossible task. Certain materials may display remarkable resistance to corrosion but exhibit high vulnerability to stress and load, while others may be highly resistant to deformation but prove unsuitable for use in acidic or salty environments [5].

It is known that the surface quality of a manufactured part has a direct impact on wear, corrosion, and the formation of cracks [6]. When a component has a poor surface finish, the likelihood of failure increases [7]. Post-processing techniques, such as heat treatment, chemical treatment, spray coating, electroplating, and electroless plating, are extensively used to improve surface strength and prevent corrosion [8], [9]. The advent of additive manufacturing has revolutionized product design and manufacturing by making it possible to produce parts with exceptional precision that were once challenging or impossible to manufacture [10]. However, the as-produced parts surface quality is often not up to standard, and post-processing of additively manufactured parts is necessary [11].

It is essential for components to possess excellent physical and chemical properties. It is difficult to achieve all the desired physical properties through a single process, so multiple processes in a suitable sequence are required [12]. In this study, we have utilized surface finishing methods to improve the surface quality of AM surfaces so that subsequent coating steps can produce the desired results. We focus on the application of chemical-based surface roughness reduction methods to additively manufactured stainless steel samples. Chemical etching methods are chosen because they effectively reduce surface roughness in hidden areas. We also report our method of producing a protective coating on the chemically polished surface of the samples. We have utilized the electroless nickel coating on additively manufactured stainless steel samples. This paper presents the experimental details of obtaining electroless nickel coatings with the state of art machine learning instance segmentation approach to analyze the microscopic and SEM images.

2. MATERIALS AND METHODS

The EOS M280 laser sintering-based metal 3D printer was utilized at the KANSAS CITY NATIONAL SECURITY CAMPUS facility to print the AM pieces. Stainless steel 316 molybdenum alloyed austenitic steel was the metal powder that was used for printing, which contains 17-19% chromium, 13-15% nickel, 2-3% molybdenum, 6-8% carbon, and the rest is iron. No information was excluded in the paraphrased text.

2.1 Sample Preparation

To prepare the samples, a multi-step process was undertaken. Initially, the samples were subjected to sonication in acetone and isopropyl alcohol to eliminate impurities such as organic and inorganic components, grease, fingerprints, light rust, tarnish, and oxides from the surface of the component. After the sonication, the samples were cleaned with a sodium hypochlorite solution at 180°F, which helped to remove the remaining solvent and oil that had been loosened in the pre-cleaning stage. Following this, the samples were electro-cleaned using a heavy-duty alkaline electrolyte from Krohn Industrial Inc. with the bath temperature maintained at 180°F and a 10V potential applied for one and a half minutes. As a result of the electro-cleaning, an oxide layer formed on the samples, which was then eliminated by submerging the samples in HCL for 40 seconds.

FIGURE 1: SAMPLE PREPARATION PROCEDURE

2.2 Electropolishing and Chempolishing

CP uses a highly concentrated acidic solution as an electrolyte which dissolves the high-stress concentration and crack nucleation regions of the sample submerged in the bath. The process is electroless, and the sample surfaces are polished after being cleaned. The chemical bath consists of 10-30% phosphoric acid, 1-10% hydrochloric acid, 1-10% nitric acid, and 1-10% surfactants that are proprietary. It is crucial to maintain the temperature of the bath at 75° C throughout the process as an increase in temperature can contaminate the chemical bath. Agitation is also a key factor in the chempolishing process, as it helps disperse the localized heat generated by the electropolishing process. To achieve this, a 20 mm magnetic stirrer was used at 200 rpm during the experiments. The samples were then rinsed in distilled water after 30 minutes of dissolution.

Electropolishing is a finishing process that utilizes electrolytic principles to remove a thin layer of material from a metal part. The process involves the use of highly concentrated acidic electrolytes that dissolve the sample continuously. In electropolishing, the electrolyte is made up of a mixture of 70% phosphoric acid and 30% sulfuric acid, while the optimal temperature for the process is 75° C. The sample and lead electrode are connected to a power source and subjected to a current density of 70 A/dm2 for 30 minutes. To neutralize the sample after electropolishing, an alkaline solution is used.

2.3 Electroless Nickel Plating

In the realm of electroless nickel plating, there are typically three types of solutions that are commonly utilized. The concentration of phosphorus within the solution is the most significant factor in determining the amount of nickel deposited. For this particular investigation, we employed electroless nickel plating solutions that were categorized as low phosphorus (ONE PLATE 3001), mid phosphorus (ONE PLATE 1001), and high phosphorus (ONE PLATE 2001). These solutions were acquired from plating international Inc. and all three are known for their stability, with pH values that fall between 5 and 6. It is vital to properly clean the sample prior to the deposition process, as surface contamination can lead to subpar deposition quality and hinder the adhesion of the Ni coating. Thus, it is essential to ensure that the substrate is free of any debris, oil, grease, and oxide layers before initiating the deposition process. To eliminate these contaminants, the sample is cleaned using acetone and isopropyl alcohol. Once this process is complete, the sample is ready to undergo activation.

To ensure the best adhesion for subsequent plating, some metals like tungsten, stainless steel, and zinc require special activation or pretreatment. Our sample was activated using a Woods nickel strike solution and 5V DC for 30 seconds. The plating process is carried out immediately after activation. It's crucial to maintain the optimal temperature during the electroless plating process, as a slight increase in temperature can cause an exothermic reaction that could damage the samples. The recommended temperature for low and medium phosphorus solution bath is 90° C, while for high phosphorus solution, it's 85° C. The deposition time for all samples was set to 30 minutes.

2.4 Taguchi Design of Experiment (TDOE)

In conducting our experiment, we utilized TDOE, which involves a series of experiments with four parameters and three levels. The first parameter pertains to the concentration of phosphorus in the electroless nickel solution, which can be classified into low, medium, and high levels. The second parameter involves the type of surface finishing applied to reduce roughness, namely EP, CP, and as-built surfaces. The third parameter concerns the 3D part coordinate plane, which affects the surface produced through selective laser melting. Our goal is to study the response of different plane surfaces to deposition. Finally, the fourth parameter is the temperature of the nickel solution, which has an optimum bath temperature for each level. We aim to observe how the nickel solution responds to temperature changes from the optimum level. We employ a temperature range of five degrees Celsius both below and above the optimum temperature.

By utilizing TDOE orthogonal arrays, we are able to reduce the number of experiments required to nine trials. This approach allows for efficient utilization of time and resources while ensuring high-quality experiments.

TABLE 1. TAGUCHI DESIGN OF EXPERIMENT

DOE	Phosphorus Content	Surface Finish	Orientation	Temperature
1	High	Elec-Pol	XY Plane	T + 5
2	High	Chem-Pol	YZ Plane	T
3	High	As-Built	XZ Plane	T - 5
4	Mid	Elec-Pol	YZ Plane	T - 5
5	Mid	Chem-Pol	XZ Plane	T + 5
6	Mid	As-Built	XY Plane	T
7	High	Elec-Pol	XZ Plane	T
8	High	Chem-Pol	XY Plane	T - 5
9	High	As-Built	YZ Plane	T + 5

2.5 Testing and Characterization

The scratch test is utilized for mechanical wear analysis and characterization, and it is crucial to apply well-defined scratches in a reproducible manner to accurately determine surfaces resistance to mechanical wear. In this study, we opted for a standard 10 N scratch test on the nickel-coated samples. To analyze the surface roughness and scratch depth, we utilized the KEYENCE Digital Microscope VHX7000, while the Phenom XL Desktop SEM was used for microstructure and elemental analysis of the nickel-deposited samples.

In order to conduct an analysis of the sample, we employed a recently developed algorithmic tool, known as "Segment Anything", which was produced by the research team at Meta AI. This tool is particularly well-suited for the task of accurately segmenting microscopic images. By using this powerful tool, we were able to effectively isolate and study the scratch within the sample in a prompt and efficient manner. Moreover, we utilized the ImageJ image processing software to precisely quantify the area of the aforementioned scratch.

3. RESULT AND DISCUSSION

The electropolishing method significantly reduce surface roughness by approximately 92%. This process quickly eliminates a considerable amount of material. Our measurements showed that the Ra value decreased from around 25 µm to 2 µm. However, this technique has more variables to consider than chempolishing, such as electrolyte concentration, current density, and sample type, which can result in uneven surface finishing. On the other hand, chempolishing offers a more uniform surface finish on all samples due to its ability to etch the component's boundaries with the same rate and ease. Nevertheless, the surface may not be entirely smooth due to the low material removal rate, resulting in an Ra value of around 11 µm after 30 minutes of chempolishing, while the as-built sample's average Ra value is approximately 25 µm.

The deposition of high phosphorus solution is generally slow due to the low nickel content of the solution, even though it results in high phosphorus concentration per deposition, which can be up to 11%. The deposition rate is approximately 5-7 µm per hour, but the plating solution is relatively stable and less sensitive to temperature changes. The coating turns the sample to amorphous structure which making it ideal for use in corrosive or acidic environments. In comparison, only a small amount of deposition is observed on the substrate for CP and as-built samples.

The study found that a nickel solution with medium levels of phosphorus results in uniform nickel deposition on all three substrates. On average, this medium phosphorus solution has a plating rate of 15 µm per hour. Analysis of the deposited sample surface reveals a phosphorus concentration of approximately 8%. This provides a viable option for plating that offers a balance between corrosion resistance and durable surface.

When applying a low phosphorus electroless nickel solution on an AM stainless steel sample, there is a significant difference compared to the as-built sample. The as-built sample has a high level of adhesion and a high concentration of nickel per deposition. On the other hand, the low phosphorus solution has an average deposition rate of 20 µm per hour.

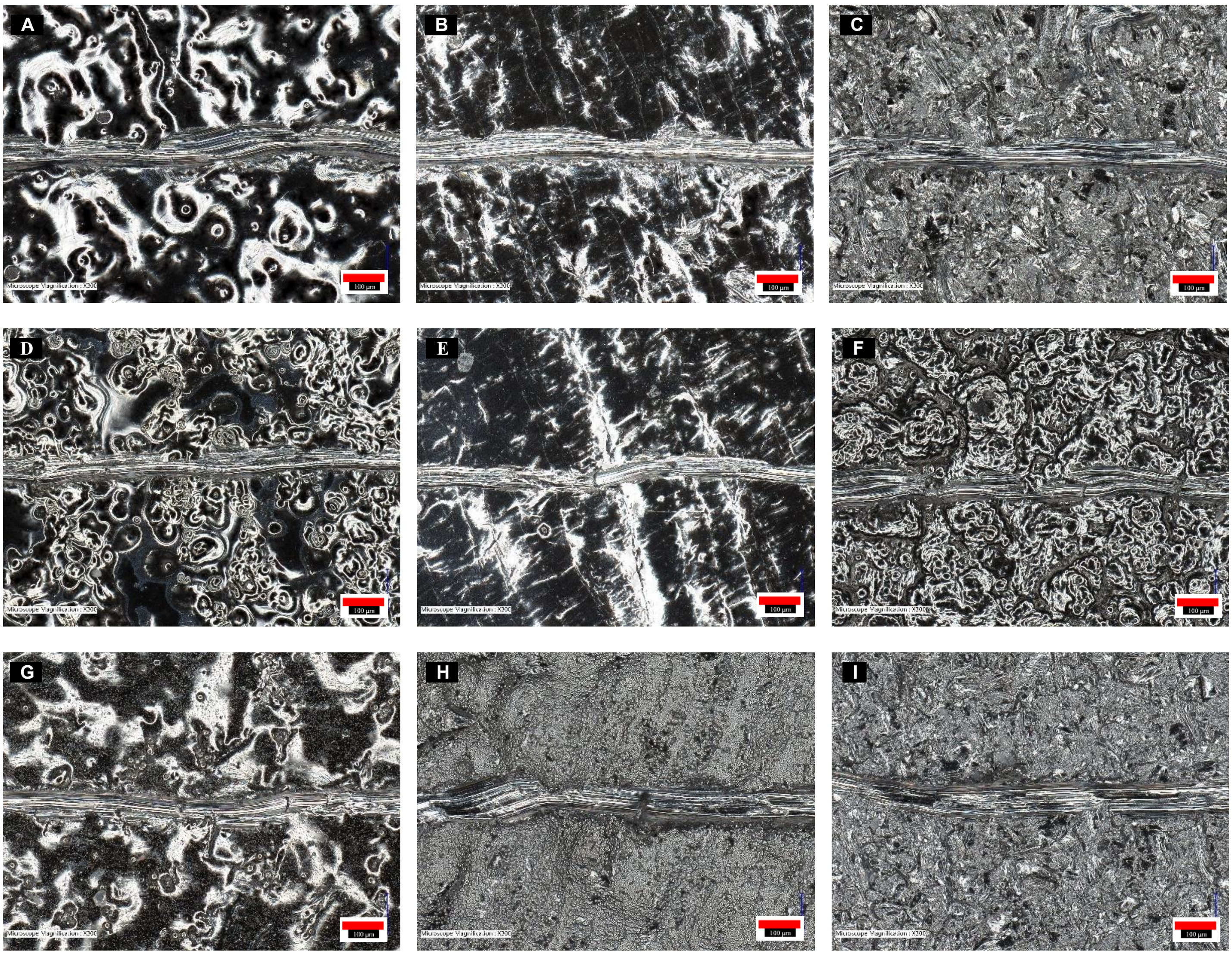

FIGURE 2: MICROSCOPY IMAGE OF SCRATCH TEST (A) DOE#1 (B) DOE#2 (C) DOE#3 (D) DOE#4 (E) DOE#5 (F) DOE#6 (G) DOE#7 (H) DOE#8 (I) DOE#9

According to the analysis of the 10 N scratch test, there was a notable improvement in scratch resistance both before and after the test. Figure 2 shows that DOE 1 and 2 experienced up to a 25% increase in scratch resistance, while DOE 3, 4,5, 7, and 8 exhibit an improvement in surface hardness ranging from 100% to 150%. DOE 6 and 9 showed the highest improvement in surface hardness which up to 197%.

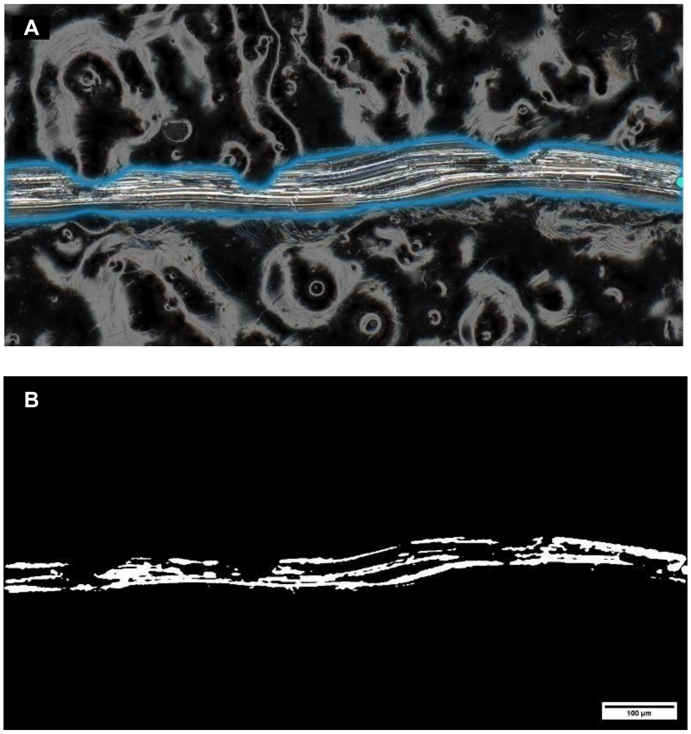

FIGURE 3: (A) INSTANCE SEGMENTATION OF MICROSCOPIC SCRATCH SAMPLE (B) IMAGEJ THRESHOLDING OF MICROSCOPIC IMAGE

Once we have performed instance segmentation and thresholding on the scratch, we proceed to determine the material's level of hardness using a formula.

$$Hs = \frac{8P}{\Pi w^2} \qquad (1)$$

Where,
Hs = Scratch hardness number (MPa)
P = Normal force (N)
w = Scratch width

The as-built sample exhibited an average surface hardness value of 290 MPa. Analysis of the hardness data indicated that the electroplated nickel sample displayed a notable increase in surface hardness, exceeding two and a half times that of the as-built sample. Specifically, DOE 6 and EOE 9 demonstrated the most pronounced enhancements in surface hardness.

Table 2. HARDNESS OF MATERIAL

DOE	Hardness of the Sample (MPa)	Improvement In hardness from As – build (%)
1	332.64	125.13
2	318.10	119.66
3	551.52	207.47
4	580.02	218.19
5	518.21	194.94
6	790.48	297.36
7	583.89	219.64
8	620.61	233.46
9	715.57	269.18

FIGURE 4: SCATTER PLOT OF HARDNESS OF THE SAMPLE AND COMPARISON OF AS BUILD SAMPLE WITH PLATED SAMPLE

4. CONCLUSION

The process of electropolishing show ability to provide high-quality surface finishing by removing material at a rapid rate. However, it does have limitations in terms of uniformity and consistency. Chempolishing, on the other hand, is a great alternative due to its ability to remove material uniformly and smooth both internal and external surfaces. Electroless nickel deposition is an excellent plating option for stainless steel samples created through additive manufacturing, as nickel offers superior wear resistance. Scratch testing has shown that nickel-plated samples are up to twice as resistant as non-plated samples. Additionally, a high-phosphorus electroless nickel solution can provide extra corrosion resistance. It should be noted that the geometry of the printed part plays a significant role in the surface finishing process. To achieve a shiny and smooth surface with excellent hardness and corrosion resistance, it may be necessary to employ successive surface finishing techniques.

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