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# STUDY OF ELECTROLESS NICKEL PLATING ON RAPID PROTOTYPING MODEL USING ACRYLONITRILE BUTADIENE STYRENE

Electroless plating on Acrylonitrile Butadiene Styrene (ABS) is a metallization process that involves a reduction and oxidation reaction between the nickel source and the substrate material. The purpose of this research is to determine the ability of nickel deposition in the nickel electroless plating process with a specific etching time variation. This nickel electroless procedure begins with a chromic acid etching process that can last anywhere from 15 to 55 minutes and is useful for increasing roughness and creating submicroscopic cavities. After the etching process is finished, the surface roughness test is performed with a Mitutoyo SJ-210. Additionally, the activation step is carried out for 5 minutes in order for the polymer to become a conductor, allowing the plating process to proceed. The electroless plating process was then carried out for 55 and 75 minutes, with the goal of depositing nickel metal on the ABS surface. The coating results were analyzed using Fourier Transform Infrared (FTIR) spectroscopy IRSpirit/ATR-S serial No. A224158/Shimadzu to determine the functional groups formed both before and after the coating process, X-Ray Diffraction (XRD) to determine the character of the crystal structure, and phase analysis of a solid material using PANalytical type E'xpert Pro, To determine the surface morphology, the Zeiss EVO MA 10 was used to perform scanning electron microscopyenergy dispersive X-ray spectroscopy (SEM-EDS) at 1000x magnification. The test findings demonstrate that, based on a range of investigations, etching variations of 15,25,35,45, and 55 minutes etching time 55 minutes are the best nickel deposited substrates, as evidenced by EDS data, where this treatment has the largest weight fraction of nickel. As a result, the longer the etching period, the rougher the surface becomes, affecting the capacity of nickel deposition to increase. Furthermore, it can be demonstrated in this investigation that the nickel deposited is in an amorphous form.

*Keywords: Electroless Nickel Plating, Acrylonitrile Butadiene Styrene, Rapid Prototyping* 

#### **1. INTRODUCTION**

Rapid prototyping began with the printing of polymers [1]. One of the most extensively utilized rapid prototyping methods in the world is fused deposition modeling (FDM) [2-4]. The dependability, safety, ease of fabrication, relatively low cost, and range of thermoplastics that can be utilized are the key reasons for this method's popularity and application. Researchers have tried to improve FDM's applicability, develop new materials, and update the FDM method itself since it was originally presented in the early 1990s [5].

ABS (acrylonitrile butadiene styrene) is a frequently used material in the FDM process [6-7]. Many industries, including aviation, automotive, biomedical, dinnerware, and others, have effectively adopted ABS. ABS plastic has intrinsic advantages that metal does not [8]. These advantages include ease of

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manufacture, light weight, and corrosion resistance. ABS's advantages do not necessarily imply that it can be employed in industrial settings. ABS and other polymers are limited by industrial demands that seek aesthetic features and some consideration of metallic properties that plastics lack, such as heat and electrical conductivity.

As a result, metallization of plastics has the potential to open up new applications for this material. Metallization is the process of converting a non-conductive material to a conductive one. The desired metallic characteristics to be present in the polymer can be delivered given the metallization procedure [10]. Copper [11], zinc [12], nickel [13], and chromium [14] are metals that can be metallized using various processes such as metal spray [15-16], metal paint [17-18], and electroless plating [19-21]. Because of the ease of the procedure, electroless plating is the most extensively utilized method among the previously stated ways in industry.

Engineers have widely recognized the applicability of electroless nickel plating due to its distinct qualities, which include strong corrosion, wear, and abrasion resistance, as well as increased ductility, impact resistance, and electrical properties [22]. The reduction of the metallic ion Ni2+ in solution and the deposition of the coating can be carried away during the oxidation of the chemicals contained in the solution itself, for example, the reducing solution, where it serves as an internal current source [23]. This procedure necessitates the reduction of the deposited Ni<sup>2+</sup> metal cations by electron acceptors, either from the surface of the metallic substance or from the surface of the catalyst employed to trigger the deposition itself.

According to the preceding description, the purpose of this study is to investigate the ability of nickel deposition on 3D printing ABS substrates with the effect of etching time.

#### 2. MATERIAL AND METHOD

#### **2.1 Material Preparation**

The first step in the material manufacturing process is to create a 3D design with dimensions of 10x10x3 mm<sup>3</sup> using SolidWorks Autodesk software. Furthermore, the CURA program is used to open the printing preparation of the constructed three-dimensional item. By altering the thickness of each layer, this software can calculate the number of layers of 3D objects to be printed.

Setting the printing parameters in the form of a nozzle angle of 60°, slicing layer of 0.2 mm, fan off speed of 40 mm/s, setting the 3D print temperature, namely the temperature at bed 100°C, nozzle temperature 250°C, and the nozzle direction longitudinally using the 3D Printer Ender 3 V2 with 100% ABS filling filament comes next.

#### 2.2 Etching Process and Surface Roughness Test After Etching Process

The first procedure was to clean ABS of various clinging contaminants for 5 minutes at 50°C using a solution of sodium carbonate and trisodium phosphate. Furthermore, the etching process was carried out with a solution of chromic acid and sulfuric acid that was heated at  $50\pm3$ °C for different processing periods. This procedure was repeated for 15, 25, 35, 45, and 55 minutes. The procedure is then followed by a neutralizing step with sodium sulfite solution. Following that, the material is immersed in room temperature hydrochloric acid for 2 minutes to remove any leftover etching solution on the plastic surface.

The process of testing surface roughness is carried out using the Mitutoyo SJ 210 Surface Roughness Tester. This test is carried out on ABS after the etching process is complete

#### 2.3 Electroless Nickel Plating

Furthermore, the activation stage to make plastic is catalytic by immersing the specimen in a solution of hydrochloric acid and palladium chloride for 5 minutes. After the ABS was cleaned with distilled water, the material was then put in a mixture of sodium hydroxide, copper sulfate and ethylene diamine tetraacetic acid disodium solution at room temperature for 5 minutes. Electroless plating is then carried out by immersing ABS in a mixture of nickel sulfate, ammonium chloride, sodium hydrogen phosphate and sodium hydroxide solution. This immersion process was carried out for 55 and 75 minutes.

#### 2.4 ABS Surface Characterization After Electroless Nickel Plating Process

In this work, X-Ray Diffraction (XRD) scanning was used to detect compounds created during the electroless nickel plating process, whereas XRD scanning was used to assess the characteristics of the ABS surface formed during the electroless nickel plating process. PANalytical type E'xpert Pro was used for XRD analysis.

Meanwhile, the IRSpirit/ATR-S serial No. A224158/Shimadzu Fourier Transform Infrared (FTIR) test was performed to learn more about the chemical bonds that exist on ABS and ABS substrates after the electroless nickel plating procedure. The distinct peaks on the ABS surface reflect the chemical bond.

A scanning electron microscope (SEM) is an electron microscope developed for analyzing the surfaces of solid objects directly. It uses a beam of concentrated electrons of relatively low energy as an electron probe that is scanned in a regular pattern over the specimen. In this work, a Zeiss EVO MA 10 scanning electron microscope (SEM) is utilized to examine the surface topology and morphology of a tested sample. Furthermore, this apparatus is equipped with Dispersive Energy X-Ray Spectroscopy (EDS) to determine a component element of a tested-sample.

#### 3. RESULTS AND DISCUSSION

Figure 1 depicts the relationship between etching time and surface roughness of 3D printed ABS. Because chemical etching with chromic acid can enhance surface roughness and increase the surface area of the substrate, the longer the etching time on the surface of acrylonitrile butadiene styrene (ABS), the roughness will increase.



Figure 1: Relationship between etching time and ABS surface roughness.

The basic idea behind this procedure is to create a hollow in the ABS substrate in order to make a link between the metal and the ABS. Furthermore, etching with chromic acid increases roughness because microscopic pores are formed when chromic acid, a strong acid, eats the surface of ABS by removing some of the surface of ABS, causing the surface roughness to increase as the etching time increases. According to the findings of the study, an etching period of 55 minutes resulted in an ABS substrate with optimal roughness, which generates acceptable conditions for nickel deposition for the electroless plating process.

The three graphs depicted in Figure 2 as XRD test results reveal that the noncrystalline solid phase is an amorphous phase. Amorphous materials are distinguished by their relatively complicated molecular structures and uneven atomic organization.



Figure 2: XRD test results on an ABS substrate

When the atomic arrangement is irregular, the intensity data that appears will tend to be unstable at adjacent angles due to reflections at different atomic densities, so that quantitative data retrieval by matching fingerprints based on the intensity and angle of the data base material cannot be carried out due to peaks. As a result, the phase of ABS and the effects of electroless nickel plating (ENP) of ABS itself can be described as amourphos.



Figure 3: FTIR analysis of ABS.

Based on Figure 3, ABS substrates, both untreated and with etching treatment of 55 minutes and electroless nickel plating 75 minutes, with analysis using FTIR resulted in the intensity of absorption characteristics at 2237, 1452, 1354, 1070 and 1028 cm<sup>-1</sup> which is a feature of ftir absorption which shows The characteristic C-N bond indicates the character of the acrylonitrile component in the copolymer. The intensity of the absorption character at 2920, 1637, 1450, 965, and 910 cm<sup>-1</sup> which shows the C-H bond which shows the characteristics of butadiene. And benzene absorption at 200-1600 cm<sup>-1</sup> which shows the styrene component. So it can be said that in electroless nickel the functional groups of the coated material do not change.



Figure 4: Surface of ABS (A) untreated (B) etching time 15 minutes ENP 55 minutes (C) etching time 55 minutes ENP

75 minutes

SEM (Scanning Electron Microscope) is used to examine the surface morphology of ABS specimens at 1000x magnification to determine the morphology on the ABS surface, where Figure 4(A) shows an ABS substrate that has not been treated, Figure 4(B) shows an ABS surface with an etching time of 15 minutes and an ENP of 55 minutes, and Figure 4(C) shows an ABS surface with an etching time of 55 minutes and an ENP 75 minutes.

As shown in Figures 4 (A) and 4 (B), the longer the etching duration, the higher the surface roughness value. This is demonstrated by the results of the SEM shot below. More scratches, cracks, and scratches from the etching results may be observed on the surface from left to right. In Figure 4 (C), where etching and electroless have been performed, the surface of the specimen will be smoother where the fractures have been deposited by nickel more than 15 minutes after etching. As a result, the surface of the specimen will be smoother where nickel has deposited fractures.

Table 1: EDS	<b>5</b> Test Results	on ABS S	pecimens.
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	Percentage (% Wt.)				
Element	Untreated	Etching time 15 min., ENP 55 min.	Etching time 55 min., ENP 75 min.		
С	87,78	84,96	82,27		
Ν	7,51	7,35	9,34		
0	2,77	5,43	4,60		
Р	1,35	1,50	1,63		
Ni	0,56	0,76	2,16		

Table 1 shows the results of EDS tests performed on ABS specimens. The electroless nickel test on ABS substrate succeeded in creating nickel deposited with the best deposition in nickel specimens with an etching time of 55, namely with a weight fraction of 2.16 percent compared to etching time 15 is 0.76 percent and without treatment with 0.56 percent, according to the results of the EDS test. Although the results from the microstructure of the etching of 15 minutes did not indicate any change in the surface contour, but with the SEM test, the etching of 15 minutes and without treatment revealed an increase in the weight fraction of nickel. The results of acid eating from the etching solution were revealed after a 15-minute etching. According to the table, the increase in P levels from no treatment is 1.35 percent etching in 15 minutes and 1.50 percent etching in 55 minutes, for a total of 1.63 percent.

However, the mechanism that occurs can be explained that electroless nickel plating is an autocatalyzed technique of deposition (reduction) of nickel ions by reducing chemicals such as hypophosphite, aminoborane, or borohydride. Hypophosphite solutions are the most widely utilized in electroless nickel processes. The mechanism of the electroless Ni-P reaction postulated in electroless nickel plating happens due to the anodic/cathodic alternate polarity that occurs in microcells on the surface of the substrate to be plated. However, the reaction can be stated as follows:

$$Ni^{2+} + H_2PO_2^- + H_2O \to Ni + H_2PO_3^- + 2H^+$$
(1)

$$3H_2PO^{2-} \rightarrow H_2PO^{2-} + H_2O + 2OH^- + 2P$$
 (2)

Reaction (1) is the one responsible for nickel reduction. While reaction (2) is in charge of phosphorus reduction.

$H_2PO_2^- + H_2O \to H^+ + HPO_3^{2-} + 2H^+$ ((	(3)	)
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$$Ni^{2+} + H_2 \rightarrow Ni + 2H^+ \tag{4}$$

$$H_2PO^{2-} + H \rightarrow P + OH^- + H_2O \tag{5}$$

 $H_2PO^{2-} + H_2O \rightarrow H^+ + HPO_3^{2-} + H_2$  (6)

$$Ni^{2+} + H_2 PO_3^{2-} \rightarrow Ni HPO_3$$
<sup>(7)</sup>

Equations (3) to (4) drive the development of Ni-P coating (5). H is generated, while Ni and P are deposited, in equations (4) and (5). Nickel precipitates as NiHPO3 as a result of Equation (7).

#### 4. CONCLUSION

Based on the research, it can be determined that the most optimum etching duration is reached at 55 minutes, which is supported by the high value of surface roughness. This has an impact on the capacity of nickel to deposit itself. The longer the etching period, the greater the nickel deposition ability, and the ensuing phase of electroless nickel plating is amorphous. Furthermore, it appears that there is a substance deposited that is assumed to be nickel and was proved using EDS with the most ideal weight fraction results in the 55 minutes etching procedure.

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