AIP Conference Proceedings

pubs.aip.org/aip/acp

Volume 2598

4th International Conference on Earth Science, Mineral and Energy

Sleman, Indonesia • 28 October 2021

Editors • Herry Riswandi, Toshifumi Igarashi, Carlito Tabelin, Timotius Pasang, Omer Faruk Ugurly, Yody Rizkianto and Oktarian Wisnu Lusantono

AIP Publishing

RESEARCH ARTICLE | JUNE 12 2023

Preface: The 4th International Conference on Earth Science, Mineral, and Energy (ICEMINE 2021) FREE

AIP Conference Proceedings 2598, 010001 (2023)
https://doi.org/10.1063/12.0014263

Preface: The 4th International Conference on Earth Science, Mineral, and Energy (ICEMINE 2021)

The 4th International Conference on Earth Science, Mineral, and Energy (ICEMINE) is an annual event that was held by Faculty of Mineral Technology, Universitas Pembangunan Nasional "Veteran" Yogyakarta. In this year, the conference brought ***"Stay Safe with Geoscience and Challenges the Mineral and Energy Exploration, Extraction and Conservation to Improve Economic Growth"*** as its theme where the discussion focused on the earth resources management to be able to improve the economic growth during and post the pandemic situation.

The conference invited six speakers across the globe where their expertise contributed enhancing the event significantly. The speakers brought six different main topics, which were disaster mitigation, groundwater characterization, clean energy transition, virtual reality utilization in the mining education, geoheritage virtual trip, and manufacturing technology. Not only that, the broad expertise from all the presenters were also enrich the discussion.

Eventually, the conference reached the goal where the pandemic could be seen as a new challenge to enhance our knowledge and technology in the earth resource management to keep the economic growing. Optimizing the existed methods, implementing a new technology, and collaboration were an important aspects to keep in mind under this hard situation. Lastly, the committee thank to everyone who made this event done succesfully.

4th International Conference on Earth Science, Mineral and Energy
AIP Conf. Proc. 2598, 010001-1–010001-1; https://doi.org/10.1063/12.0014263
Published by AIP Publishing. 978-0-7354-4543-7/$30.00

RESEARCH ARTICLE | JUNE 12 2023

Effect of nickel electroplating process time variation over aluminum on crystalplane orientation FREE

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AIP Conference Proceedings 2598, 040009 (2023)
https://doi.org/10.1063/5.0126191

Effect Of Nickel Electroplating Process Time Variation Over Aluminum On CrystalPlane Orientation

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Abstract. Metal plating starts to be widely used as a way to prevent metal corrosion. Apart from preventing corrosion, metal coatings were being developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. The purpose of this research is to make Ni coating over Al substrate in time variation differences (3.5 h, 4.5 h and 5 h) with a constant current of 50 mA cm-2. The characterization of the materials used was from SEM-EDS, XRD and digital cameras. The SEM characterization results showed a crystal shape similar to a pyramid and contained Ni oxide content in the Ni coating, although in the XRD observations did not appear. The increasing of the coating time will increase the crystal size in the direction of the preferred plane orientation
[111] which was confirmed by the calculation of the orientation index. The Ni 5 sample has hydrophobic properties with a contact angle of 91 °.

Keywords: electrodeposition,hydrophobic,SEM

INTRODUCTION

Aluminum metal is one of the materials that is widely used by people such as transportation, buildings and construction as well as home accessories and decorations because of its soft and stylish appearance, its color is silvery and shiny. Prices are quite affordable and easy to obtain, different uses of aluminum metal in the engineering field The use of aluminum metal in the engineering field is easy to form, a good heat conductor, resistant to cold temperatures, and aluminum can also be corroded. [1], [2]

Metal plating is starting to be widely used as a way to prevent metal from corrosion. In addition to preventing corrosion, metal coatings have been developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. [3] - [6]. Many factors affect to get this benefits results which are the composition of the material, the composition of the solution [7], [8], the temperature of the solution [9] - [12], the current density of the solution [13] - [15], the duration of immersion and the electric voltage [5], [13] - [20]. The electroplatingprocess has long been recognized for plating metals such as nickel, chrome, gold and silver. Electroplating method offers many advantages such as faster, reliable and relatively cheaper [21] [22] [23] - [26].

The crystal size has a direct impression on the surface energy of the coating. Along with surface energy, surface morphology also plays a vital role in surface wettability. Because of the change in current density (j), thickness,layer morphology and crystal size can be obtained. Therefore, this research will look for changes in the microstructureof the layer and the distribution of Ni particles on the Al substrate with time variations [27], [28].

Research conducted by Zubar yo et al using electrolyte solutions of $NiSO_4.6H_2O$ (210g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) [29]. This solution is also called a watts solution because it uses three main supporting components: nickel sulfate, nickel chloride, and boric acid.

4th International Conference on Earth Science, Mineral and Energy
AIP Conf. Proc. 2598, 040009-1–040009-8; https://doi.org/10.1063/5.0126191
Published by AIP Publishing. 978-0-7354-4543-7/$30.00

Although research on Nickel plating on aluminum substrates has been done a lot. Eventhough many studies of nickel-to-aluminum plating with time variations have been carried out, there are still many lack of details regarding the effect of nickel plating on aluminum by the electrodeposition method on crystal size, surface morphology, statisticalcrystal distribution and wettability. Material characterization in this study using SEM, XRD and digital cameras.

METHOD

The anode rod used is 98% pure nickel metal **Table 2** and the cathode is an aluminum substrate **Table 1**. The electrolyte solution used was a watts solution of $NiSO_4.6H_2O$ (210g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) with quality / grade analyst from the chemical manufacturer Merck. Before the experiment, the aluminum metal was polished and washed in an ultrasonic bath. The watts solution to be used is treated first by stirring for 1 hour with a magnetic stirred (Bante Instrument MS 3000) at a speed of 200 rpm to get homogeneity. The electroplating experiment used a current of 50 mA cm-2 with a time variation of 3.5 hours, 4.5 hours and 5 hours.

TABLE 1. Chemical composition of the Al alloy substrates (wt%), determined from the X-ray fluorescence measurement

Element	Al	Mg	Fe
Concentration	96,88	1,49	1,63

Each sample will be written as Ni 3.5, Ni 4.5 and Ni 5 to represent each experiment with time variation in this study. The anode rod used in this research has the following composition:

TABLE 2. Chemical composition of the pure Ni (wt%), determined from the X-ray fluorescence measurement.

Element	**Concentration (%)**
Al (Alumunium)	0,02
Ca (Kalsium)	0,04
Fe (Besi)	0,23
Ni (Nikel)	**98,01**
Y (Yitrium)	1,61
Zr (Zirkon)	0,04
Nb (Niobium)	0,05

Surface morphological analysis of the nickel plating experiment on aluminum using FE-SEM (FE-SEMFEI INSPECT F50 EDAX EDS Analyzer) with magnifications of 2500 × and 10000 ×. The crystalline structure of the Nickel layer was identified by XRD (Rigaku RINT 2000 with Cu K radiation). XRD data is processed with the help of HighScore Plus software to refined and calculate the crystal size, besides that, the position of the particles can also be known.

Water contact angle observations were taken using a Canon 1000D EOS camera. The value determines criteria of the angle θ as $\theta < 90°$, $90° \leq \theta < 150°$, $150° \leq \theta < 180°$ for being hydrophilic, hydrophobic, and superhydrophobic, respectively [28]

RESULT AND DISCUSSION

Electroplating is used to form a microstructural pattern of nickel plating over aluminum. The time variation will provide information about changes in the shape of the surface morphology and affect the size of the crystals(3.5 h, 4.5 and 5). In addition, we can determine the crystal size distribution using statistical analysis with ImageJ (Oppen Access) and Origin (OriginLab Corporation). By measuring the length of the grain diameter by ImageJ, then processing it by Origin.

Figure 1 shows the microstructure evolution of pure Nickel plating over aluminum with time variations. It can be seen from the SEM results that Nickel is evenly distributed over the Aluminum substrate in the electroplating process with a time of 3.5 h (**Figure 1a**). Then it can be seen visually from the increase in time to 4.5 h the crystal size is getting bigger and looks quite close to each other (**Figure 1b**). A customarily distributed Gaussian pattern can confirm this. Finally, at the time variation of 5 h, the crystals became visually more compact and bonded to one another (**Figure 1c**).

As additional information, it can be seen that all layers of Ni 3.5 - Ni 5 (**Figure. 1 a - c**), showed a half pyramidal structure with a crystal (8 - 30 µm) [30]. This pyramidal structure is morphologically typical for Ni plating with apreferred plane texture in a plane. [100,111] [31] - [33].

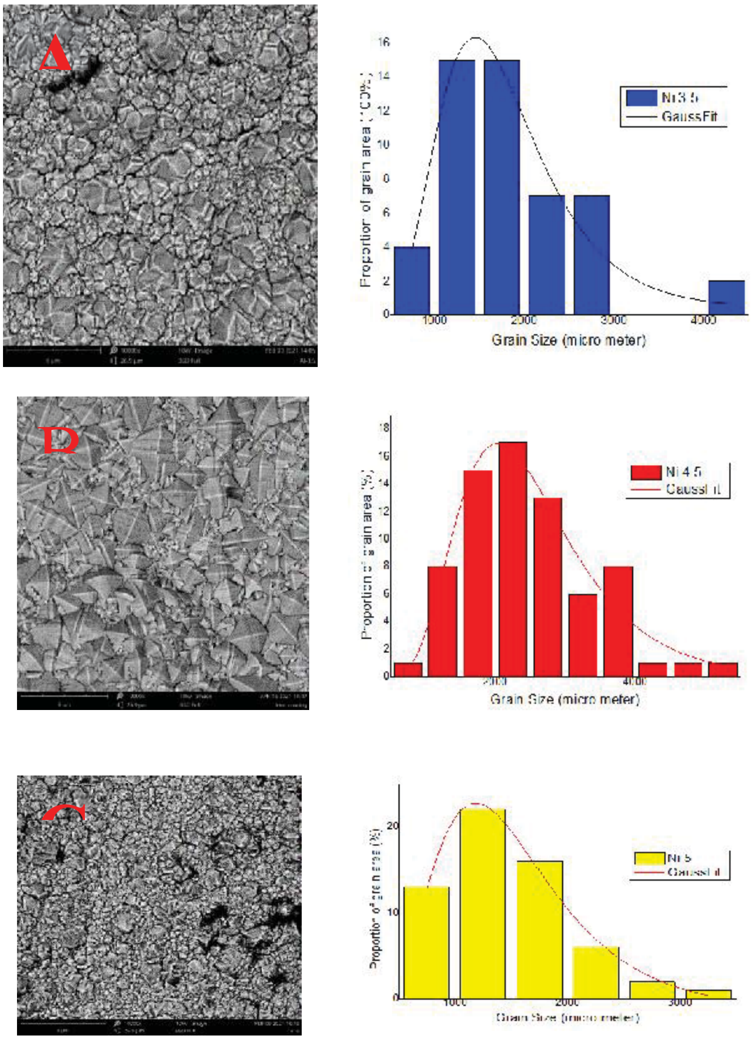

FIGURE 1 (a-c). SEM micrographs for different layers show the morphology of pure Ni coating separately deposited and distribution particle obtained using ImageJ combine Origin with corresponding Gauss fitting function

In the electroplating process, an anodizing event occurs. The cathode attracts the positive ions because a unidirectional electric current flows between the two electrodes in the electrolyte solution. Apart from releasing positive ions, the anode electrode also releases oxygen. Oxygen gas needs to be considered because as an inhibitor for anode electrode to releases positive ions [34], [35].

The presence of oxygen gas can be seen with the EDS pattern of nickel electroplating on an aluminum substrate presented in **Figure 2**. EDS analyzes the presence of significant peaks of nickel and oxygen peaks.

Transferring positive ions from the anode to the cathode causes a small amount of carbon to accumulate in the Ni anode rod. Readings of how much the weight percent Ni, C and O2 can be seen in table 3.

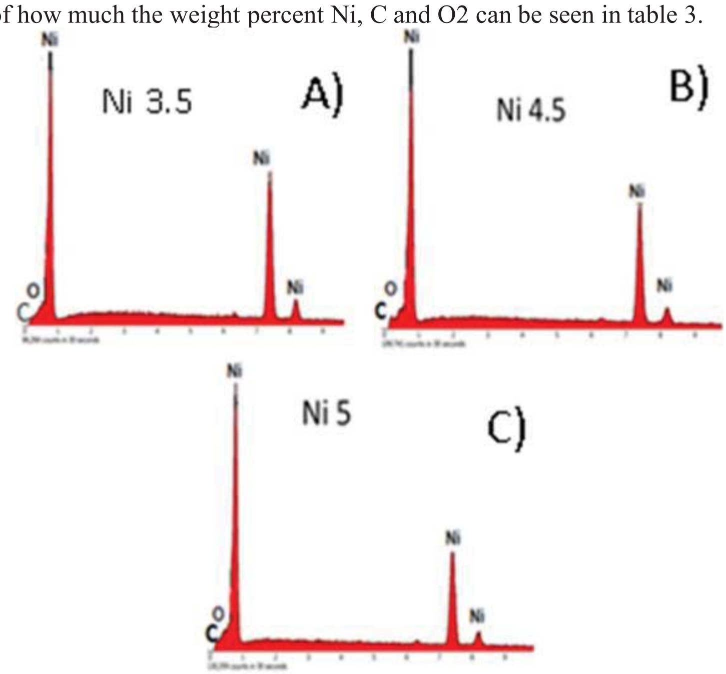

FIGURE 2. EDS pattern showing an element of Ni, C and O at various times

TABLE 3. Ni, C and O content on electrodeposit Ni Film with different time

Sampel	Ni (wt%)	C (wt%)	O (wt%)
Ni 3.5	98.30	0.90	0.80
Ni 4.5	96.86	2.00	1.14
Ni 5	95.47	2.41	2.12

When viewed from the data in table 3, it can be seen that the Ni 3.5 sample weight percent Ni 98.30 (wt%),C 0.90 (wt%) and O2 0.80 (wt%). Then for the sample Ni 5 has a weight percent Ni 95.47 (wt%), C 2.00 (wt%) and O2 1.14 (wt%). It can conclud that the increasing variety of time will increase the weight percent (wt%) of O2. the increase in O2 comes from oxidized Ni, but even though EDS detected O2, it was not observed by XRD

The calculation of the μ orientation index aims to determine the crystal structure growing or even shrinking along with the electroplating process with time variations. The orientation index μ (hkl) is calculated using the equation:

$$\mu(hkl) = \frac{I(hkl)/I_0(hkl)}{\frac{1}{n}\sum I(h^F k^F l^F)/I_0(h^F k^F l^F)}$$

Where is the peak intensity of each crystal plane from the research data, while is the peak intensity of the crystalis obtained from the ICSD (Inorganic Crystal Structure Database). In addition, $I(h'k'l')$ is the summation of the three peak intensities from the research data and $I_0(h'k'l')$ is the summation of the three peak intensities from the ICSD data. An index value greater than 1 $[\mu(hkl) \geq 1]$ indicates that the crystal field (*hkl*) is growing, meanwhile, if $[\mu(hkl) \leq 1]$ indicates that the crystal field is shrinking.

TABLE 4. Calculation of automatic index for coating Ni over Al with time variation

Sample	hkl			μ(hkl)		
	[111]	[002]	[022]	[111]	[002]	[022]
Ni 3.5	3702	1950	668	0,70	1,18	1,08
Ni 4.5	8570	967	619	1,63	0,58	1,00
Ni 5	3466	2006	557	0,66	1,22	0,90

In this study, the Ni over Al coating with the current density was kept constant at 50 mA cm^{-2} with timevariations showing that the crystal plane grew and shrank from the calculation of the orientation index μ.In the sample Ni 4.5, the crystal plane [111] known to have a value of 1.63, which means that compared to Ni 3.5, the crystal plane grows more due to the addition of time. Crystal field [022] experienced growth in the Ni 3.5 and Ni 5 samples, but shrank in the Ni5 samples. This is due to a decrease in electrical conductivity from the use of solution concentration..

The time variation effect affects the growth orientation of a particular crystal plane. The longer the plating time will affect the orientation of a certain crystal plane. This is estimated from the higher concentration of Ni^{2+} ions in solution. The growth of plane orientation [111] has advantages including anti-corrosion, increasing electrical conductivity and increasing mechanical properties [36].

The results of the plane orientation in table 4 are confirmed by the XRD pattern produced. The XRD pattern is shown in **Figure 3** results from the linear refinement of the XRD pattern processed using the HighscorePlus Software. The crystal structure formed is a face centered cubic (FCC) which shows three significant peaks for the plane [111], [002] and [022]. The phase formed is a single phase and no other phases are formed on the surface of the Ni layer. The results of XRD data processing did not show nickel oxide, even though O_2 was detected by EDS (**Figure 2**), because the oxide formed in Ni was small.

There are three significantly different peak intensities, the peaks are the plane [111], [002] and[022]. The difference in peak intensity is due to the difference in mass density on the surface of each crystal plane due to time variations.

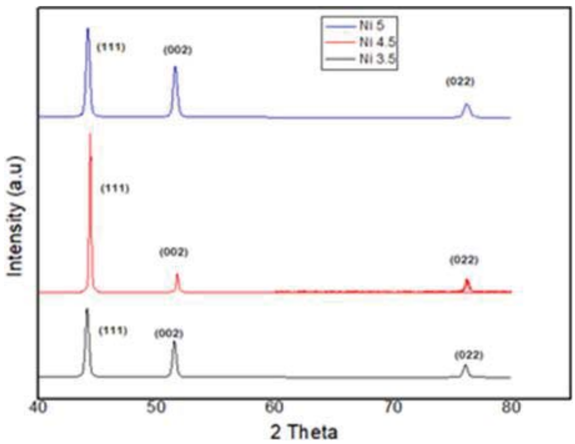

FIGURE 3. XRD pattern of electrodeposit Ni onto Al substrate with various time at constant current densities 50mA cm^{-2}

The calculation of crystal size uses the Debye-Scherrer equation, where k is a constant of 0.9, β (the width of the diffraction peak of FWHM in radians, λ (the x-ray wavelength using Cu Kα radiation is 0.154nm), and θ (Bragg's angle) [37].

$$D = \frac{k}{\beta \cos\theta}$$

The crystal parameters of the sample after doing Rietvel Refinement using HighScore Plus software arepresented in table 5. Rietveld corrections were made until the GOF (Goodness of fit) value was less than2.5% and the ROP

(R-weighted pattern) value was less than 10%. The atom's position can also be seen in the table which states that the atom's position occupies every point in the cube with its occupancy value of 1, so it can be concluded that there is no atomic shift.

TABLE 5. Crystal parameter of Ni films at various times and constant current densities at 50mA cm^{-2}

Parameter		Sample		
		Ni 3.5	Ni 4.5	Ni 5
Crystal structure		Cubic FCC	Cubic FCC	Cubic FCC
Space Group		Fm-3m	Fm-3m	Fm-3m
space Group Number		225	225	225
Laticce constan (Å)a = b = c		3,5175	3,5206	3,5152
Volume ($Å_3$)		44.52	43,64	43,44
Densitas (g/cm3)		8,79	8,88	8,79
d-spacing (Å)		1,0142	1,0093	2,04906
Crystallite Size Debye-Scherrer(nm)		24	42	23
Atomic Position	X	0	0	0
	Y	0	0	0
	Z	0	0	0
	Occupancy	1	1	1
R-weighted pattern		7,67075	9,09029	7,249
GOF (Goodness of Fit)		0,74151	1,00618	1,986

Determination of hydrophilic and hydrophobic properties can be done using a water drop test over the sample. **Figure 4** presents a photo of the results of the drop test that has been carried out. In the drop test, it can be seen that there is a difference in contact angle due to time variations. Therefore, it can be concluded that over time the angle of contact increases as well. This has also been observed by [38] in their study. Samples Ni 3.5 and Ni 4.5 show hydrophilic properties because the contact angle is 73 ° and 88 °. Ni 5 sample has hydrophobic properties because it has a contactangle of 91 ° where the range for hydrophobic properties (90 ° ≤ θ <150 °) [38].

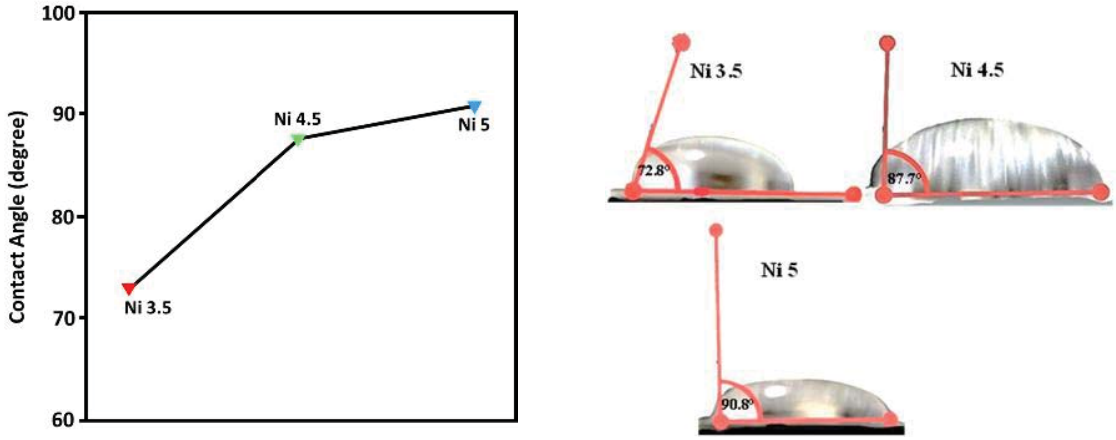

Figure 4: Wettability of the various Ni films deposited on the Al alloy substrates at different time.

CONCLUSION AND RECOMMENDATION

Nickel plating has been successfully carried out on the Al substrate with time variations. The effect of Ni coating affects surface morphology, crystal orientation, crystallite size and wettability. The Ni 4.5 sample has the largest crystal size of 44nm with the most dominant crystal plane [111] this is also confirmed by calculating the orientation index, this means that Ni elektropalting on aluminium with time variations has been successfully carried out. The morphological structure of the crystal is in the form of a pyramid completely distributed over the Al substrate as seen from the Gaussian plot. In the Ni 5 sample has hydrophobic properties with a contact angle of91 °. Even though the EDS characterization contained Ni oxide, but in XRD observations were not found because theO2 that formed was very small

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Submission date: 31-Mar-2024 01:27AM (UTC+0700)
Submission ID: 2207546240
File name: 34._Cahaya_Rosyidan.pdf (524.46K)
Word count: 3911
Character count: 20695

RESEARCH ARTICLE | JUNE 12 2023

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AIP Conference Proceedings 2598, 040009 (2023)
https://doi.org/10.1063/5.0126191

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Abstract. Metal plating starts to be widely used as a way to prevent metal corrosion. Apart from preventing corrosion, metal coatings were being developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. The purpose of this research is to make Ni coating over Al substrate in time variation differences (3.5 h, 4.5 h and 5 h) with a constant current of 50 mA cm-2. The characterization of the materials used was from SEM-EDS, XRD and digital cameras. The SEM characterization results showed a crystal shape similar to a pyramid and contained Ni oxide content in the Ni coating, although in the XRD observations did not appear. The increasing of the coating time will increase the crystal size in the direction of the preferred plane orientation

[111] which was confirmed by the calculation of the orientation index. The Ni 5 sample has hydrophobic properties with a contact angle of 91 °.

Keywords: electrodeposition,hydrophobic,SEM

INTRODUCTION

Aluminum metal is one of the materials that is widely used by people such as transportation, buildings and construction as well as home accessories and decorations because of its soft and stylish appearance, its color is silvery and shiny. Prices are quite affordable and easy to obtain, different uses of aluminum metal in the engineering field The use of aluminum metal in the engineering field is easy to form, a good heat conductor, resistant to cold temperatures, and aluminum can also be corroded. [1], [2]

Metal plating is starting to be widely used as a way to prevent metal from corrosion. In addition to preventing corrosion, metal coatings have been developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. [3] - [6]. Many factors affect to get this benefits results which are the composition of the material, the composition of the solution [7], [8], the temperature of the solution [9] - [12], the current density of the solution [13] - [15], the duration of immersion and the electric voltage [5], [13] - [20]. The electroplatingprocess has long been recognized for plating metals such as nickel, chrome, gold and silver. Electroplating method offers many advantages such as faster, reliable and relatively cheaper [21] [22] [23] - [26].

The crystal size has a direct impression on the surface energy of the coating. Along with surface energy, surface morphology also plays a vital role in surface wettability. Because of the change in current density (j), thickness,layer morphology and crystal size can be obtained. Therefore, this research will look for changes in the microstructureof the layer and the distribution of Ni particles on the Al substrate with time variations [27], [28].

Research conducted by Zubar yo et al using electrolyte solutions of $NiSO_4.6H_2O$ (210g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) [29]. This solution is also called a watts solution because it uses three main supporting components: nickel sulfate, nickel chloride, and boric acid.

4th International Conference on Earth Science, Mineral and Energy
AIP Conf. Proc. 2598, 040009-1–040009-8; https://doi.org/10.1063/5.0126191
Published by AIP Publishing. 978-0-7354-4543-7/$30.00

Although research on Nickel plating on aluminum substrates has been done a lot. Eventhough many studies of nickel-to-aluminum plating with time variations have been carried out, there are still many lack of details regarding the effect of nickel plating on aluminum by the electrodeposition method on crystal size, surface morphology, statisticalcrystal distribution and wettability. Material characterization in this study using SEM, XRD and digital cameras.

METHOD

The anode rod used is 98% pure nickel metal **Table 2** and the cathode is an aluminum substrate **Table 1**. The electrolyte solution used was a watts solution of $NiSO_4.6H_2O$ (210g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) with quality / grade analyst from the chemical manufacturer Merck. Before the experiment, the aluminum metal was polished and washed in an ultrasonic bath. The watts solution to be used is treated first by stirring for 1 hour with a magnetic stirred (Bante Instrument MS 3000) at a speed of 200 rpm to get homogeneity. The electroplating experiment used a current of 50 mA cm-2 with a time variation of 3.5 hours, 4.5 hours and 5 hours.

TABLE 1. Chemical composition of the Al alloy substrates (wt%), determined from the X-ray fluorescence measurement

Element	Al	Mg	Fe
Concentration	96,88	1,49	1,63

Each sample will be written as Ni 3.5, Ni 4.5 and Ni 5 to represent each experiment with time variation in this study. The anode rod used in this research has the following composition:

TABLE 2. Chemical composition of the pure Ni (wt%), determined from the X-ray fluorescence measurement.

Element	**Concentration (%)**
Al (Alumunium)	0,02
Ca (Kalsium)	0,04
Fe (Besi)	0,23
Ni (Nikel)	**98,01**
Y (Yitrium)	1,61
Zr (Zirkon)	0,04
Nb (Niobium)	0,05

Surface morphological analysis of the nickel plating experiment on aluminum using FE-SEM (FE-SEMFEI INSPECT F50 EDAX EDS Analyzer) with magnifications of 2500 × and 10000 ×. The crystalline structure of the Nickel layer was identified by XRD (Rigaku RINT 2000 with Cu K radiation). XRD data is processed with the help of HighScore Plus software to refined and calculate the crystal size, besides that, the position of the particles can also be known.

Water contact angle observations were taken using a Canon 1000D EOS camera. The value determines criteria of the angle θ as $\theta < 90°$, $90° \leq \theta < 150°$, $150° \leq \theta < 180°$ for being hydrophilic, hydrophobic, and superhydrophobic, respectively [28]

RESULT AND DISCUSSION

Electroplating is used to form a microstructural pattern of nickel plating over aluminum. The time variation will provide information about changes in the shape of the surface morphology and affect the size of the crystals(3.5 h, 4.5 and 5). In addition, we can determine the crystal size distribution using statistical analysis with ImageJ (Oppen Access) and Origin (OriginLab Corporation). By measuring the length of the grain diameter by ImageJ, then processing it by Origin.

Figure 1 shows the microstructure evolution of pure Nickel plating over aluminum with time variations. It can be seen from the SEM results that Nickel is evenly distributed over the Aluminum substrate in the electroplating process with a time of 3.5 h (**Figure 1a**). Then it can be seen visually from the increase in time to 4.5 h the crystal size is getting bigger and looks quite close to each other (**Figure 1b**). A customarily distributed Gaussian pattern can confirm this. Finally, at the time variation of 5 h, the crystals became visually more compact and bonded to one another (**Figure 1c**).

As additional information, it can be seen that all layers of Ni 3.5 - Ni 5 (**Figure. 1 a - c**), showed a half pyramidal structure with a crystal (8 - 30 μm) [30]. This pyramidal structure is morphologically typical for Ni plating with apreferred plane texture in a plane. [100,111] [31] - [33].

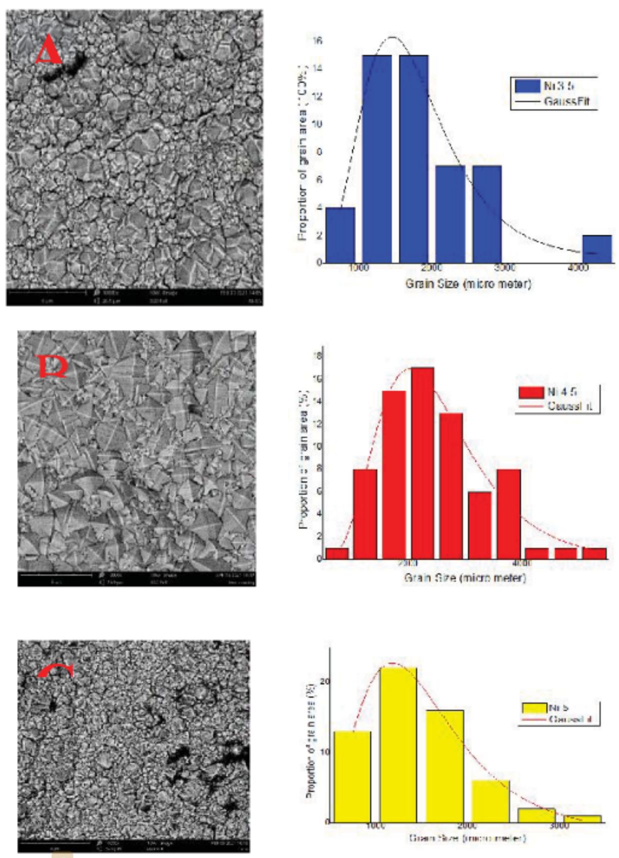

FIGURE 1 (a-c). SEM micrographs for different layers show the morphology of pure Ni coating separately deposited and distribution particle obtained using ImageJ combine Origin with corresponding Gauss fitting function

In the electroplating process, an anodizing event occurs. The cathode attracts the positive ions because a unidirectional electric current flows between the two electrodes in the electrolyte solution. Apart from releasing positive ions, the anode electrode also releases oxygen. Oxygen gas needs to be considered because as an inhibitor for anode electrode to releases positive ions [34], [35].

The presence of oxygen gas can be seen with the EDS pattern of nickel electroplating on an aluminum substrate presented in **Figure 2**. EDS analyzes the presence of significant peaks of nickel and oxygen peaks.

Transferring positive ions from the anode to the cathode causes a small amount of carbon to accumulate in the Ni anode rod. Readings of how much the weight percent Ni, C and O2 can be seen in table 3.

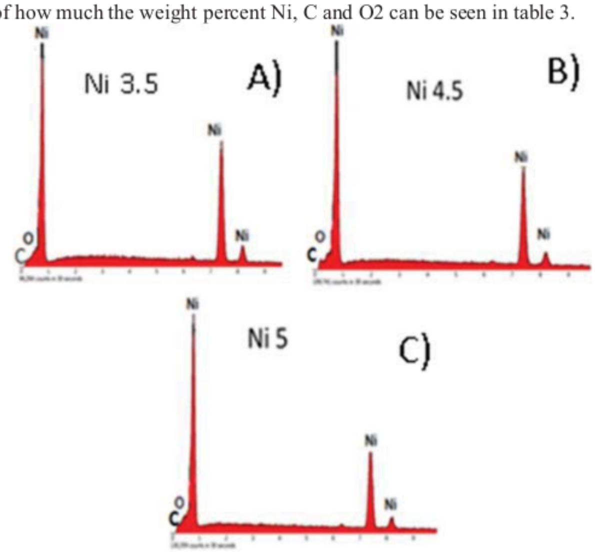

FIGURE 2. EDS pattern showing an element of Ni, C and O at various times

TABLE 3. Ni, C and O content on electrodeposit Ni Film with different time

Sampel	Ni (wt%)	C (wt%)	O (wt%)
Ni 3.5	98.30	0.90	0.80
Ni 4.5	96.86	2.00	1.14
Ni 5	95.47	2.41	2.12

When viewed from the data in table 3, it can be seen that the Ni 3.5 sample weight percent Ni 98.30 (wt%),C 0.90 (wt%) and O2 0.80 (wt%). Then for the sample Ni 5 has a weight percent Ni 95.47 (wt%), C 2.00 (wt%) and O2 1.14 (wt%). It can conclud that the increasing variety of time will increase the weight percent (wt%) of O2. the increase in O2 comes from oxidized Ni, but even though EDS detected O2, it was not observed by XRD

The calculation of the μ orientation index aims to determine the crystal structure growing or even shrinking along with the electroplating process with time variations. The orientation index μ (hkl) is calculated using the equation:

$$\mu(hkl) = \frac{I(hkl)/I_0(hkl)}{\frac{1}{n}\sum I(h'k'l')/I_0(h'k'l')}$$

Where is the peak intensity of each crystal plane from the research data, while is the peak intensity of the crystalis obtained from the ICSD (Inorganic Crystal Structure Database). In addition, $I(h'k'l')$ is the summation of the three peak intensities from the research data and $I_0(h'k'l')$ is the summation of the three peak intensities from the ICSD data. An index value greater than 1 [$\mu(hkl) \geq 1$] indicates that the crystal field (*hkl*) is growing, meanwhile, if [$\mu(hkl) \leq 1$] indicates that the crystal field is shrinking.

TABLE 4. Calculation of automatic index for coating Ni over Al with time variation

Sample	hkl			μ(hkl)		
	[111]	[002]	[022]	[111]	[002]	[022]
Ni 3.5	3702	1950	668	0,70	1,18	1,08
Ni 4.5	8570	967	619	1,63	0,58	1,00
Ni 5	3466	2006	557	0,66	1,22	0,90

In this study, the Ni over Al coating with the current density was kept constant at 50 mA cm^{-2} with timevariations showing that the crystal plane grew and shrank from the calculation of the orientation index μ.In the sample Ni 4.5, the crystal plane [111] known to have a value of 1.63, which means that compared to Ni 3.5, the crystal plane grows more due to the addition of time. Crystal field [022] experienced growth in the Ni 3.5 and Ni 5 samples, but shrank in the Ni5 samples. This is due to a decrease in electrical conductivity from the use of solution concentration..

The time variation effect affects the growth orientation of a particular crystal plane. The longer the plating time will affect the orientation of a certain crystal plane. This is estimated from the higher concentration of Ni^{2+} ions in solution. The growth of plane orientation [111] has advantages including anti-corrosion, increasing electrical conductivity and increasing mechanical properties [36].

The results of the plane orientation in table 4 are confirmed by the XRD pattern produced. The XRD pattern is shown in **Figure 3** results from the linear refinement of the XRD pattern processed using the HighscorePlus Software. The crystal structure formed is a face centered cubic (FCC) which shows three significant peaks for the plane [111], [002] and [022]. The phase formed is a single phase and no other phases are formed on the surface of the Ni layer. The results of XRD data processing did not show nickel oxide, even though O_2 was detected by EDS (**Figure 2**), because the oxide formed in Ni was small.

There are three significantly different peak intensities, the peaks are the plane [111], [002] and[022]. The difference in peak intensity is due to the difference in mass density on the surface of each crystal plane due to time variations.

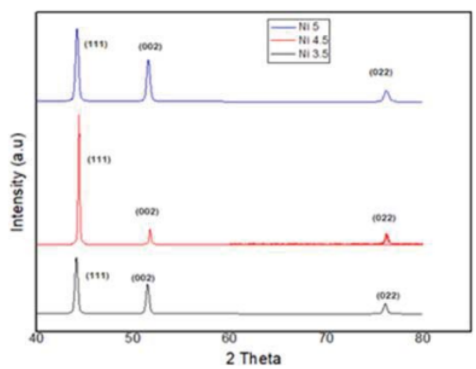

FIGURE 3. XRD pattern of electrodeposit Ni onto Al substrate with various time at constant current densities 50mA cm^{-2}

The calculation of crystal size uses the Debye-Scherrer equation, where k is a constant of 0.9, β (the width of the diffraction peak of FWHM in radians, λ (the x-ray wavelength using Cu Kα radiation is 0.154nm), and θ (Bragg's angle) [37].

$$D = \frac{k}{\beta \cos \theta}$$

The crystal parameters of the sample after doing Rietvel Refinement using HighScore Plus software arepresented in table 5. Rietveld corrections were made until the GOF (Goodness of fit) value was less than2.5% and the ROP

(R-weighted pattern) value was less than 10%. The atom's position can also be seen in the table which states that the atom's position occupies every point in the cube with its occupancy value of 1, so it can be concluded that there is no atomic shift.

TABLE 5. Crystal parameter of Ni films at various times and constant current densities at 50mA cm^{-2}

Parameter		Sample		
		Ni 3.5	Ni 4.5	Ni 5
Crystal structure		Cubic FCC	Cubic FCC	Cubic FCC
Space Group		Fm-3m	Fm-3m	Fm-3m
space Group Number		225	225	225
Laticce constan (Å)a = b = c		3,5175	3,5206	3,5152
Volume (Å$_3$)		44.52	43,64	43,44
Densitas (g/cm3)		8,79	8,88	8,79
d-spacing (Å)		1,0142	1,0093	2,04906
Crystallite Size Debye-Scherrer(nm)		24	42	23
Atomic Position	X	0	0	0
	Y	0	0	0
	Z	0	0	0
	Occupancy	1	1	1
R-weighted pattern		7,67075	9,09029	7,249
GOF (Goodness of Fit)		0,74151	1,00618	1,986

Determination of hydrophilic and hydrophobic properties can be done using a water drop test over the sample. **Figure 4** presents a photo of the results of the drop test that has been carried out. In the drop test, it can be seen that there is a difference in contact angle due to time variations. Therefore, it can be concluded that over time the angle of contact increases as well. This has also been observed by [38] in their study. Samples Ni 3.5 and Ni 4.5 show hydrophilic properties because the contact angle is 73 ° and 88 °. Ni 5 sample has hydrophobic properties because it has a contactangle of 91 ° where the range for hydrophobic properties (90 ° ≤ θ <150 °) [38].

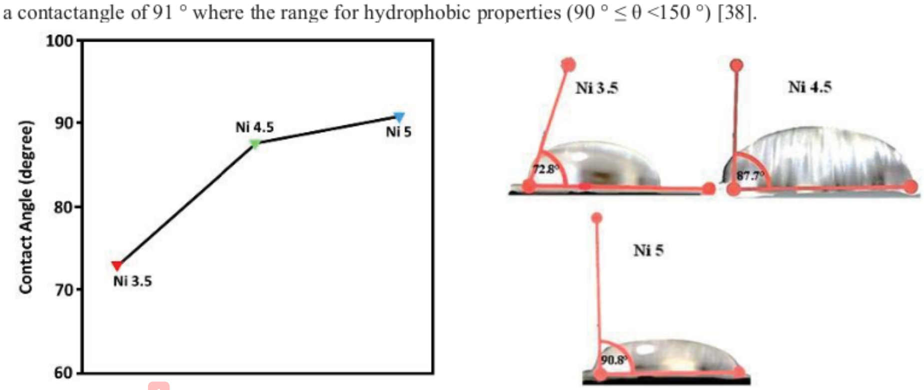

Figure 4: Wettability of the various Ni films deposited on the Al alloy substrates at different time.

CONCLUSION AND RECOMMENDATION

Nickel plating has been successfully carried out on the Al substrate with time variations. The effect of Ni coating affects surface morphology, crystal orientation, crystallite size and wettability. The Ni 4.5 sample has the largest crystal size of 44nm with the most dominant crystal plane [111] this is also confirmed by calculating the orientation index, this means that Ni elektropalting on aluminium with time variations has been successfully carried out. The morphological structure of the crystal is in the form of a pyramid completely distributed over the Al substrate as seen from the Gaussian plot. In the Ni 5 sample has hydrophobic properties with a contact angle of91 °. Even though the EDS characterization contained Ni oxide, but in XRD observations were not found because theO_2 that formed was very small

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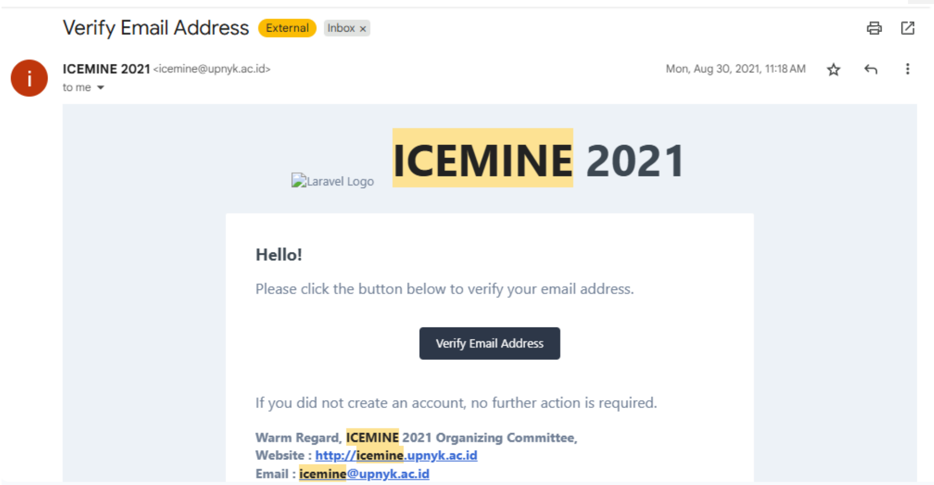

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EFFECT OF NICKEL ELECTROPLATING PROCESS TIME VARIATION OVER ALUMINUM ON CRYSTALPLANE ORIENTATION

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Abstract. Metal plating is starting to be widely used as a way to prevent metal corrosion. Apart from preventing corrosion, metal coatings were being developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. The purpose of this research is to make Ni over Al coating in time variation differences (3.5 h, 4.5 h and 5 h) with a constant current of 50 mA cm-

2. The characterization of the materials used was from SEM-EDS, XRD and digital cameras. The SEM characterization results showed a crystal shape similar to a pyramid and contained Ni oxide content in the Ni coating, although in the XRD observations did not appear. The increasing of the coating time will increase the crystal size in the direction of the preferred plane orientation

[111] which was confirmed by the calculation of the orientation index. The Ni 5 sample has hydrophobic properties with a contact angle of 91 °.

Keywords: electrodeposition,hydrophobic,
SEM

1.1. INTRODUCTION

Aluminum metal is one of the materials that is widely used by people such as transportation, buildings and construction as well as home accessories and decorations because of its soft and stylish appearance, its color is silvery and shiny. Prices are quite affordable and easy to obtain, different uses of aluminum metal in the engineering field The use of aluminum metal in the engineering field is easy to form, a good heat conductor, resistant to cold temperatures, and aluminum can also be corroded. [1], [2]

Metal plating is starting to be widely used as a way to prevent metal from corrosion. In addition to preventing corrosion, metal coatings have been developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. [3] - [6]. Many factors affect to get this benefits results which are the composition of the material, the composition of the solution [7], [8], the temperature of the solution [9] - [12], the current strength ofthe solution [13] - [15], the duration of immersion and the electric voltage solutions [5], [13] - [20]. The electroplatingprocess has long been recognized for plating metals such as nickel, chrome, gold and silver. Electroplating method offers many advantages such as faster, reliable and relatively cheaper [21] [22] [23] - [26].

The crystal size has a direct impression on the surface energy of the coating. Along with surface energy, surface morphology also plays a vital role in surface wettability. Because of the change in current density (j), thickness,layer morphology and crystal size can be obtained. Therefore, this research will look for changes in the microstructureof the layer and the distribution of Ni particles on the Al substrate with time variations [27], [28].

Research conducted by Zubar yo et al using electrolyte solutions of $NiSO_4.6H_2O$ (210 g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) [29]. This solution is also called a watts solution because it uses three main supporting components: nickel sulfate, nickel chloride, and boric acid.

Although research on Nickel plating on aluminum substrates has been done a lot. Eventhough many studies of nickel-to-aluminum plating with time variations have been carried out, there are still many lack of details regarding the effect of nickel plating on aluminum by the electrodeposit method on crystal size, surface morphology, statistical crystal distribution and wettability. Material characterization in this study using SEM, XRD and digital cameras.

1.2. METHOD

The anode rod used is 98% pure nickel metal **Table 2** and cathode is an aluminum substrate **Table 1**. The electrolyte solution used was a watts solution of $NiSO_4.6H_2O$ (210 g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) with quality / grade analyst from the chemical manufacturer Merck. Before the experiment, the aluminum metal was polished and washed in an ultrasonic bath. The watts solution to be used is treated first by stirring for 1 hour with a magnetic stirred (Bante Instrument MS 3000) at a speed of 200 rpm to get homogeneity. The electroplating experiment used a current of 50 mA cm-2 with a time variation of 3.5 hours, 4.5 hours and 5 hours.

TABLE 1. Chemical composition of the Al alloy substrates (wt%), determined from the X-ray fluorescence measurement

Element	Al	Mg	Fe
Concentration	96,88	1,49	1,63

Each sample will be written as Ni 3.5, Ni 4.5 and Ni 5 to represent each experiment with time variation in this study. The anode rod used in this research has the following composition:

TABLE 2. Chemical composition of the pure Ni (wt%), determined from the X-ray fluorescence measurement.

Element	**Concentration (%)**
Al (Alumunium)	0,02
Ca (Kalsium)	0,04
Fe (Besi)	0,23
Ni (Nikel)	**98,01**
Y (Yitrium)	1,61
Zr (Zirkon)	0,04
Nb (Niobium)	0,05

Surface morphological analysis of the nickel plating experiment on aluminum using FE-SEM (FE-SEMFEI INSPECT F50 EDAX EDS Analyzer) with magnifications of 2500 × and 10000 ×. The crystalline structure of the Nickel layer was identified by XRD (Rigaku RINT 2000 with Cu K radiation). XRD data is processed with the help of HighScore Plus software to refined and calculate the crystal size, besides that, the position of the particles can also be known.

Water contact angle observations were taken using a Canon 1000D EOS camera. The value determines criteria of the angle θ as $\theta < 90°$, $90° \leq \theta < 150°$, $150° \leq \theta < 180°$ for being hydrophilic, hydrophobic, and superhydrophobic, respectively [28]

1.3. RESULT AND DISCUSSION

Electroplating is used to form a microstructural pattern of nickel plating over aluminum. The time variation will provide information about changes in the shape of the surface morphology and affect the size of the crystals(3.5 h, 4.5 and 5). In addition, we can determine the crystal size distribution using statistical analysis with ImageJ (Oppen Access) and Origin (OriginLab Corporation). By measuring the length of the grain diameter by ImageJ, then processing it by Origin.

Figure 1 shows the microstructure evolution of pure Nickel plating over aluminum with time variations. It can be seen from the SEM results that Nickel is evenly distributed over the Aluminum substrate in the electroplating process with a time of 3.5 h (**Figure 1a**). Then it can be seen visually from the increase in time to 4.5 h the crystal size is getting bigger and looks quite close to each other (**Figure 1b**). A customarily distributed Gaussian pattern can confirm this. Finally, at the time variation of 5 h, the crystals became visually more compact and bonded to one another (**Figure 1c**).

As additional information, it can be seen that all layers of Ni 3.5 - Ni 5 (**Figure. 1 a - c**), showed a half pyramidal structure with a grain area (8 - 30 µm) [30]. This pyramidal structure is morphologically typical for Ni plating with a preferred plane texture in a plane. [100,111] [31] - [33].

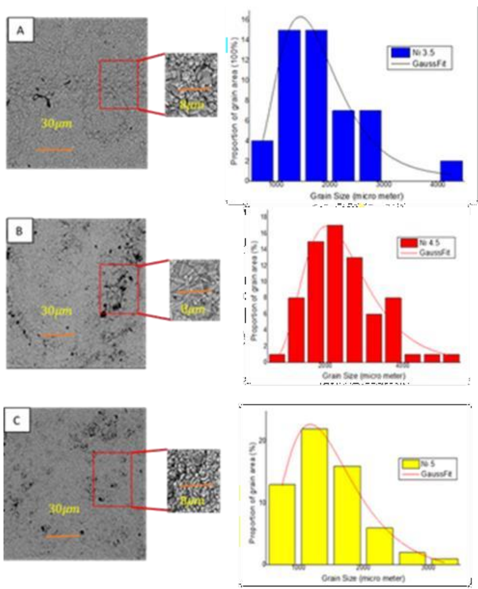

FIGURE 1 (a-c). SEM micrographs for different layers show the morphology of pure Ni coating separately deposited and distribution particle obtained using ImageJ combine Origin with corresponding Gauss fitting function

In the electroplating process, an anodizing event occurs. The cathode attracts the positive ions because a unidirectional electric current flows between the two electrodes in the electrolyte solution. Apart from releasing positive ions, the anode electrode also releases oxygen. Oxygen gas needs to be considered because as an inhibitor for anode electrode to releases positive ions [34], [35].

The presence of oxygen gas can be seen with the EDS pattern of nickel electroplating on an aluminum substrate presented in **Figure 2**. EDS analyzes the presence of significant peaks of nickel and oxygen peaks.

Transferring positive ions from the anode to the cathode causes a small amount of carbon to accumulate in the Ni anode rod. Readings of how much the weight percent Ni, C and O2 can be seen in table 3.

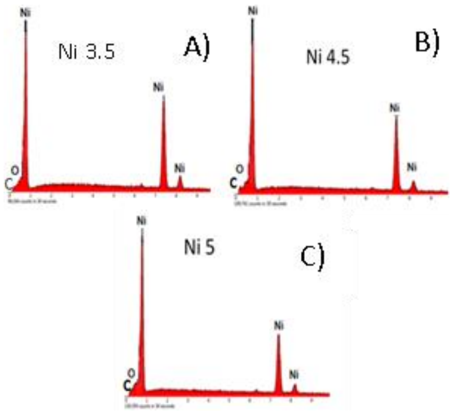

FIGURE 2. EDS pattern showing an element of Ni, C and O at various times

TABLE 3. Ni, C and O content on electrodeposit Ni Film with different time

Sampel	Ni (wt%)	C (wt%)	O (wt%)
Ni 3.5	98.30	0.90	0.80
Ni 4.5	96.86	2.00	1.14
Ni 5	95.47	2.41	2.12

When viewed from the data in table 3, it can be seen that the Ni 3.5 sample weight percent Ni 98.30 (wt%),C 0.90 (wt%) and O2 0.80 (wt%). Then for the sample Ni 5 has a weight percent Ni 95.47 (wt%), C 2.00 (wt%) and O2 1.14 (wt%). It can conclud that the increasing variety of time will increase the weight percent (wt%) of O2. the increase in O2 comes from oxidized Ni, but even though EDS detected O2, it was not observed by XRD

The calculation of the µ orientation index aims to determine the crystal structure growing or even shrinking along with the electroplating process with time variations. The orientation index µ (hkl) is calculated using the equation:

$$\mu\ (hkl) = \frac{I(hkl)/I_0(hkl)}{\frac{1}{n}\sum I(h^F k^F l^F)/I_0\ (h^F k^F l^F)}$$

Where is the peak intensity of each crystal plane from the research data, while is the peak intensity of the crystalis obtained from the ICSD (Inorganic Crystal Structure Database). In addition, $I(h'k'l')$ is the summation of the three peak intensities from the research data and $I_0(h'k'l')$ is the summation of the three peak intensities from the ICSD data. An index value greater than 1 $[\mu(hkl) \geq 1]$ indicates that the crystal field (hkl) is growing, meanwhile, if $[\mu(hkl) \leq 1]$ indicates that the crystal field is shrinking.

TABLE 4. Calculation of automatic index for coating Ni over Al with time variation

Sample	hkl			μ(hkl)		
	[111]	[002]	[022]	[111]	[002]	[022]
Ni 3.5	3702	1950	668	0,70	1,18	1,08
Ni 4.5	8570	967	619	1,63	0,58	1,00
Ni 5	3466	2006	557	0,66	1,22	0,90

1.3.1. In this study, the Ni over Al coating with the current density was kept constant at 50 mA cm^{-2} with timevariations showing that the crystal plane grew and shrank from the calculation of the orientation index µ.In the sample Ni 4.5, the crystal plane [111] known to have a value of 1.63, which means that compared to Ni 3.5, the crystal plane grows more due to the addition of time. Crystal field [022] experienced growth in the Ni 3.5 and Ni 5 samples, but shrank in the Ni5 samples. This is due to a decrease in electrical conductivity from the use of solution concentration..

The time variation effect affects the growth orientation of a particular crystal plane. The longer the plating time will affect the orientation of a certain crystal plane. This is estimated from the higher concentration of Ni^{2+} ions in solution. The growth of plane orientation [111] has advantages including anti-corrosion, increasing electrical conductivity and increasing mechanical properties [36].

1.3.2. The results of the plane orientation in table 4 are confirmed by the XRD pattern produced. The XRD pattern is shown in Figure 3 results from the linear refinement of the XRD pattern processed using the HighscorePlus Software. The crystal structure formed is a face centered cubic (FCC) which shows three significant peaks for the plane [111], [002] and [022]. The phase formed is a single phase and no other phases are formed on the surface of the Ni layer. The results of XRD data processing did not show nickel oxide, even though O_2 was detected by EDS (Figure 2), because the oxide formed in Ni was small.

There are three significantly different peak intensities, the peaks are the plane [111], [002] and[022]. The difference in peak intensity is due to the difference in mass density on the surface of each crystal plane due to time variations.

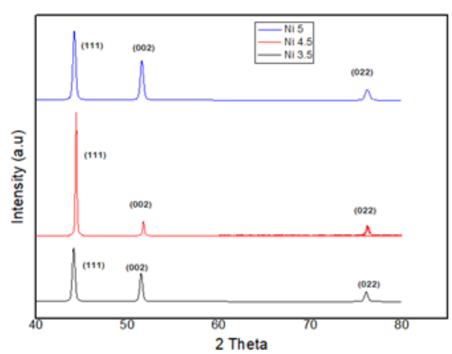

FIGURE 3. XRD pattern of electrodeposit Ni onto Al substrate with various time at constant current densities 50mA cm^{-2}

1.3.3. The calculation of crystal size uses the Debye-Scherrer equation, where k is a constant of 0.9, β (the width of the diffraction peak of FWHM in radians, λ (the x-ray wavelength using Cu Kα radiation is 0.154nm), and θ (Bragg's angle) [37].

$$D = \frac{k\lambda}{\beta \cos\theta}$$

1.3.4. The crystal parameters of the sample after doing Rietvel Refinement using HighScore Plus software arepresented in table 5. Rietveld corrections were made until the GOF (Goodness of fit) value was less than2.5% and the ROP (R-weighted pattern) value was less than 10%. The atom's position can also be seen in the table which states that the atom's position occupies every point in the cube with its occupancy value of 1, so it can be concluded that there is no atomic shift.

TABLE 5. Crystal parameter of Ni films at various times and constant current densities at 50mA cm^{-2}

Parameter		**Sample**		
		Ni 3.5	**Ni 4.5**	**Ni 5**
Crystal structure		Cubic FCC	Cubic FCC	Cubic FCC
Space Group		Fm-3m	Fm-3m	Fm-3m
space Group Number		225	225	225
Laticce constan (Å) a = b = c		3,5175	3,5206	3,5152
Volume (Å$_3$)		44.52	43,64	43,44
Densitas (g/cm3)		8,79	8,88	8,79
d-spacing (Å)		1,0142	1,0093	2,04906
Crystallite Size Debye-Scherrer (nm)		24	42	23
Atomic Position	X	0	0	0
	Y	0	0	0
	Z	0	0	0
	Occupancy	1	1	1
R-weighted pattern		7,67075	9,09029	7,249
GOF (Goodness of Fit)		0,74151	1,00618	1,986

Determination of hydrophilic and hydrophobic properties can be done using a water drop test over the sample. **Figure 4** presents a photo of the results of the drop test that has been carried out. In the drop test, it can be seen that there is a difference in contact angle due to time variations. Therefore, it can be concluded that over time the angle of contact increases as well. This has also been observed by [38] in their study. Samples Ni 3.5 and Ni 4.5 show hydrophilic properties because the contact angle is 73 ° and 88 °. Ni 5 sample has hydrophobic properties because it has a contactangle of 91 ° where the range for hydrophobic properties (90 ° ≤ θ <150 °) [38].

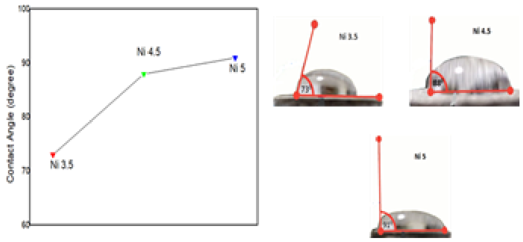

Figure 4: Wettability of the various Ni films deposited on the Al alloy substrates at different time..

1.4. CONCLUSION AND RECOMMENDATION

Nickel plating has been successfully carried out on the Al substrate with time variations. The effect of Ni coating affects surface morphology, crystal orientation, crystallite size and wettability. The Ni 4.5 sample has the largest crystal size of 44nm with the most dominant crystal plane [111] this is also confirmed by calculating the orientation index, this mean that Ni electroplating on aluminium with time variations has been successfully carried out. The morphological structure of the crystal is in the form of a pyramid completely distributed over the Al substrate as seen from the Gaussian plot. In the Ni 5 sample has hydrophobic properties with a contact angle of91 °. Even though the EDS characterization contained Ni oxide, but in XRD observations were not found because theO2 that formed was very small

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EFFECT OF NICKEL ELECTROPLATING PROCESS TIME VARIATION OVER ALUMINUM ON CRYSTALPLANE ORIENTATION

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2.1. INTRODUCTION

Aluminum metal is one of the materials that is widely used by people such as transportation, buildings and construction as well as home accessories and decorations because of its soft and stylish appearance, its color is silvery and shiny. Prices are quite affordable and easy to obtain, different uses of aluminum metal in the engineering field The use of aluminum metal in the engineering field is easy to form, a good heat conductor, resistant to cold temperatures, and aluminum can also be corroded. [1], [2]

Metal plating is starting to be widely used as a way to prevent metal from corrosion. In addition to preventing corrosion, metal coatings have been developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. [3] - [6]. Many factors affect to get this benefits results which are the composition of the material, the composition of the solution [7], [8], the temperature of the solution [9] - [12], the current density of the solution [13] - [15], the duration of immersion and the electric voltage [5], [13] - [20]. The electroplatingprocess has long been recognized for plating metals such as nickel, chrome, gold and silver. Electroplating method offers many advantages such as faster, reliable and relatively cheaper [21] [22] [23] - [26].

The crystal size has a direct impression on the surface energy of the coating. Along with surface energy, surface morphology also plays a vital role in surface wettability. Because of the change in current density (j), thickness,layer morphology and crystal size can be obtained. Therefore, this research will look for changes in the microstructureof the layer and the distribution of Ni particles on the Al substrate with time variations [27], [28].

Research conducted by Zubar yo et al using electrolyte solutions of $NiSO_4.6H_2O$ (210g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) [29]. This solution is also called a watts solution because it uses three main supporting components: nickel sulfate, nickel chloride, and boric acid.

Although research on Nickel plating on aluminum substrates has been done a lot. Eventhough many studies of nickel-to-aluminum plating with time variations have been carried out, there are still many lack of details regarding the effect of nickel plating on aluminum by the electrodeposition method on crystal size, surface morphology, statisticalcrystal distribution and wettability. Material characterization in this study using SEM, XRD and digital cameras.

2.2. METHOD

The anode rod used is 98% pure nickel metal **Table 2** and the cathode is an aluminum substrate **Table 1**. The electrolyte solution used was a watts solution of $NiSO_4.6H_2O$ (210g L^{-1}), $NiCl_2.6H_2O$ (20 gL^{-1}) and H_3BO_3 (45 g L^{-1}) with quality / grade analyst from the chemical manufacturer Merck. Before the experiment, the aluminum metal was polished and washed in an ultrasonic bath. The watts solution to be used is treated first by stirring for 1 hour with a magnetic stirred (Bante Instrument MS 3000) at a speed of 200 rpm to get homogeneity. The electroplating experiment used a current of 50 mA cm^{-2} with a time variation of 3.5 hours, 4.5 hours and 5 hours.

Commented [U1]: What the solution?

TABLE 3. Chemical composition of the Al alloy substrates (wt%), determined from the X-ray fluorescence measurement

Element	Al	Mg	Fe
Concentration	96,88	1,49	1,63

Each sample will be written as Ni 3.5, Ni 4.5 and Ni 5 to represent each experiment with time variation in this study. The anode rod used in this research has the following composition:

TABLE 4. Chemical composition of the pure Ni (wt%), determined from the X-ray fluorescence measurement.

Element	Concentration (%)
Al (Alumunium)	0,02
Ca (Kalsium)	0,04
Fe (Besi)	0,23
Ni (Nikel)	**98,01**
Y (Yitrium)	1,61
Zr (Zirkon)	0,04
Nb (Niobium)	0,05

Surface morphological analysis of the nickel plating experiment on aluminum using FE-SEM (FE-SEMFEI INSPECT F50 EDAX EDS Analyzer) with magnifications of 2500 × and 10000 ×. The crystalline structure of the Nickel layer was identified by XRD (Rigaku RINT 2000 with Cu K radiation). XRD data is processed with the help of HighScore Plus software to refined and calculate the crystal size, besides that, the position of the particles can alsobe known.

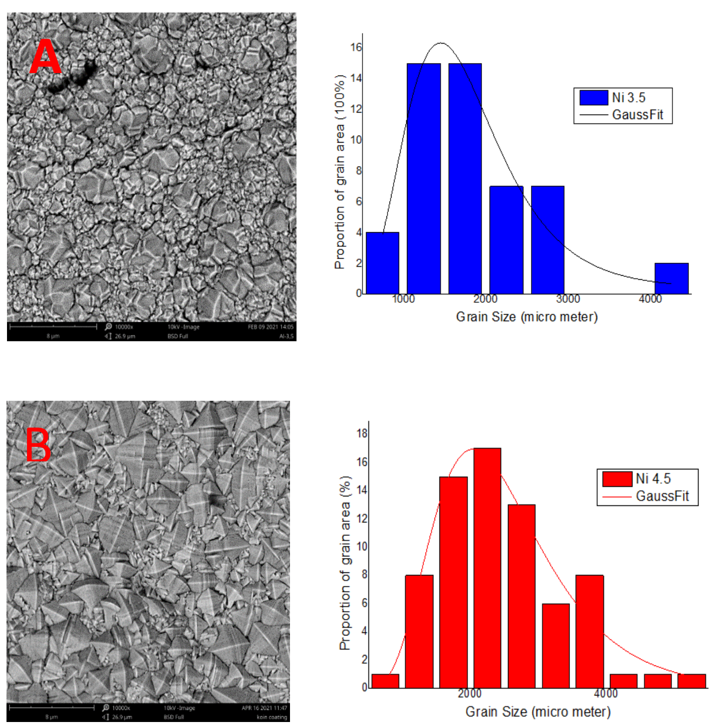
Water contact angle observations were taken using a Canon 1000D EOS camera. The value determines criteria of the angle θ as $\theta < 90°$, $90° \leq \theta < 150°$, $150° \leq \theta < 180°$ for being hydrophilic, hydrophobic, and superhydrophobic, respectively [28]

2.3. RESULT AND DISCUSSION

Electroplating is used to form a microstructural pattern of nickel plating over aluminum. The time variation will provide information about changes in the shape of the surface morphology and affect the size of the crystals (3.5 h, 4.5 and 5). In addition, we can determine the crystal size distribution using statistical analysis with ImageJ (Oppen Access) and Origin (OriginLab Corporation). By measuring the length of the grain diameter by ImageJ, then processing it by Origin.

Figure 1 shows the microstructure evolution of pure Nickel plating over aluminum with time variations. It can beseen from the SEM results that Nickel is evenly distributed over the Aluminum substrate in the electroplating processwith a time of 3.5 h (**Figure 1a**). Then it can be seen visually from the increase in time to 4.5 h the crystal size is getting bigger and looks quite close to each other (**Figure 1b**). A customarily distributed Gaussian pattern can confirm this. Finally, at the time variation of 5 h, the crystals became visually more compact and bonded to one another (**Figure 1c**).

As additional information, it can be seen that all layers of Ni 3.5 - Ni 5 (**Figure. 1 a - c**), showed a half pyramidal structure with a crystal (8 - 30 µm) [30]. This pyramidal structure is morphologically typical for Ni plating with apreferred plane texture in a plane. [100,111] [31] - [33].



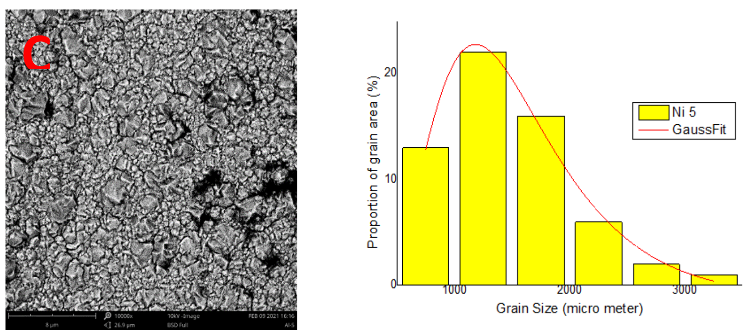


FIGURE 3 (a-c). SEM micrographs for different layers show the morphology of pure Ni coating separately deposited and distribution particle obtained using ImageJ combine Origin with corresponding Gauss fitting function

In the electroplating process, an anodizing event occurs. The cathode attracts the positive ions because a unidirectional electric current flows between the two electrodes in the electrolyte solution. Apart from releasing positive ions, the anode electrode also releases oxygen. Oxygen gas needs to be considered because as an inhibitor for anode electrode to releases positive ions [34], [35].

The presence of oxygen gas can be seen with the EDS pattern of nickel electroplating on an aluminum substrate presented in **Figure 2**. EDS analyzes the presence of significant peaks of nickel and oxygen peaks.

Transferring positive ions from the anode to the cathode causes a small amount of carbon to accumulate in the Ni anode rod. Readings of how much the weight percent Ni, C and O_2 can be seen in table 3.

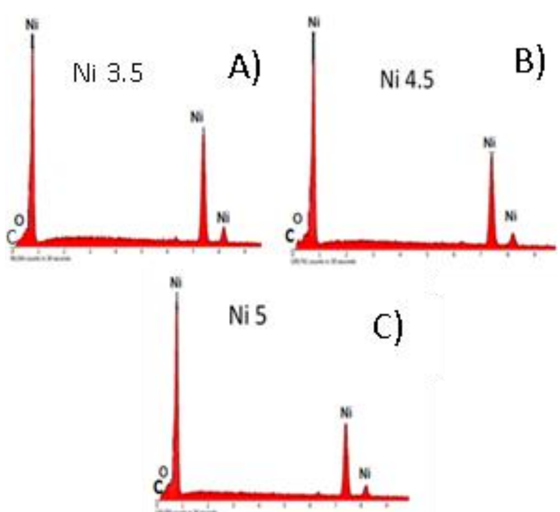


FIGURE 4. EDS pattern showing an element of Ni, C and O_2 at various times

TABLE 3. Ni, C and O_2 content on electrodeposit Ni Film with different time

Sampel	Ni (wt%)	C (wt%)	O_2 (wt%)
Ni 3.5	98.30	0.90	0.80
Ni 4.5	96.86	2.00	1.14
Ni 5	95.47	2.41	2.12

When viewed from the data in table 3, it can be seen that the Ni 3.5 sample weight percent Ni 98.30 (wt%),C 0.90 (wt%) and O_2 0.80 (wt%). Then for the sample Ni 5 has a weight percent Ni 95.47 (wt%), C 2.00 (wt%) and O_2 1.14 (wt%). It can conclud that the increasing variety of time will increase the weight percent (wt%) of O_2. the increase in O_2 comes from oxidized Ni, but even though EDS detected O_2, it was not observed by XRD

The calculation of the μ orientation index aims to determine the crystal structure growing or even shrinking alongwith the electroplating process with time variations. The orientation index μ (hkl) is calculated using the equation:

$$\mu\ (hkl) = \frac{I(hkl)/I_0(hkl)}{\frac{1}{n}\sum I(h^F k^F l^F)/I_0(h^F k^F l^F)}$$

Where is the peak intensity of each crystal plane from the research data, while is the peak intensity of the crystal is obtained from the ICSD (Inorganic Crystal Structure Database). In addition, $I(h'k'l')$ is the summation of the threepeak intensities from the research data and $I_0(h'k'l')$ is the summation of the three peak intensities from the ICSD data. An index value greater than 1 $[\mu(hkl) \geq 1]$ indicates that the crystal field (*hkl*) is growing, meanwhile, if $[\mu(hkl) \leq 1]$ indicates that the crystal field is shrinking.

TABLE 4. Calculation of automatic index for coating Ni over Al with time variation

hkl	μ(hkl)

Sample	[111]	[002]	[022]	[111]	[002]	[022]
Ni 3.5	3702	1950	668	0,70	1,18	1,08
Ni 4.5	8570	967	619	1,63	0,58	1,00
Ni 5	3466	2006	557	0,66	1,22	0,90

2.3.1. In this study, the Ni over Al coating with the current density was kept constant at 50 mA cm^{-2} with timevariations showing that the crystal plane grew and shrank from the calculation of the orientation index µ.In the sample Ni 4.5, the crystal plane [111] known to have a value of 1.63, which means that compared to Ni 3.5, the crystal plane grows more due to the addition of time. Crystal field [022] experienced growth in the Ni 3.5 and Ni 5 samples, but shrank in the Ni5 samples. This is due to a decrease in electrical conductivity from the use of solution concentration.

The time variation effect affects the growth orientation of a particular crystal plane. The longer the plating time will affect the orientation of a certain crystal plane. This is estimated from the higher concentration of Ni^{2+} ions in solution. The growth of plane orientation [111] has advantages including anti-corrosion, increasing electrical conductivity and increasing mechanical properties [36].

2.3.2. The results of the plane orientation in table 4 are confirmed by the XRD pattern produced. The XRD pattern is shown in Figure 3 results from the linear refinement of the XRD pattern processed using the HighscorePlus Software. The crystal structure formed is a face centered cubic (FCC) which shows three significant peaks for the plane [111], [002] and [022]. The phase formed is a single phase and no other phases are formed on the surface of the Ni layer. The results of XRD data processing did not show nickel oxide, even though O_2 was detected by EDS (Figure 2), because the oxide formed in Ni was small.

There are three significantly different peak intensities, the peaks are the plane [111], [002] and[022]. The difference in peak intensity is due to the difference in mass density on the surface of each crystal plane due to time variations.

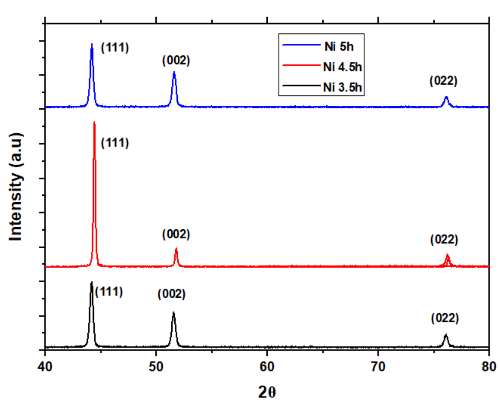


FIGURE 3. XRD pattern of electrodeposit Ni onto Al substrate with various time at constant current densities 50mA cm^{-2}

2.3.3. The calculation of crystal size uses the Debye-Scherrer equation, where k is a constant of 0.9, β (the width of the diffraction peak of FWHM in radians, λ (the x-ray wavelength using Cu Kα radiation is 0.154nm), and θ (Bragg's angle) [37].

$$D = \frac{k\lambda}{\beta \cos\theta}$$

2.3.4. The crystal parameters of the sample after doing Rietvel Refinement using HighScore Plus software arepresented in table 5. Rietveld corrections were made until the GOF (Goodness of fit) value was less than2.5% and the ROP (R-weighted pattern) value was less than 10%. The atom's position can also be seen in the table which states that the atom's position occupies every point in the cube with its occupancy value of 1, so it can be concluded that there is no atomic shift.

TABLE 5. Crystal parameter of Ni films at various times and constant current densities at 50mA cm^{-2}

Parameter		**Sample**		
		Ni 3.5	**Ni 4.5**	**Ni 5**
Crystal structure		Cubic FCC	Cubic FCC	Cubic FCC
Space Group		Fm-3m	Fm-3m	Fm-3m
space Group Number		225	225	225
Laticce constan (Å)a = b = c		3,5175	3,5206	3,5152
Volume (Å$_3$)		44.52	43,64	43,44
Densitas (g/cm3)		8,79	8,88	8,79
d-spacing (Å)		1,0142	1,0093	2,04906
Crystallite Size Debye-Scherrer(nm)		24	42	23
Atomic Position	X	0	0	0
	Y	0	0	0
	Z	0	0	0
	Occupancy	1	1	1
R-weighted pattern		7,67075	9,09029	7,249
GOF (Goodness of Fit)		0,74151	1,00618	1,986

Determination of hydrophilic and hydrophobic properties can be done using a water drop test over the sample. **Figure 4** presents a photo of the results of the drop test that has been carried out. In the drop test, it can be seen that there is a difference in contact angle due to time variations. Therefore, it can be concluded that over time the angle of contact increases as well. This has also been observed by [38] in their study. Samples Ni 3.5 and Ni 4.5 show hydrophilic properties because the contact angle is 73 ° and 88 °. Ni 5 sample has hydrophobic properties because it has a contactangle of 91 ° where the range for hydrophobic properties (90 ° ≤ θ <150 °) [38].

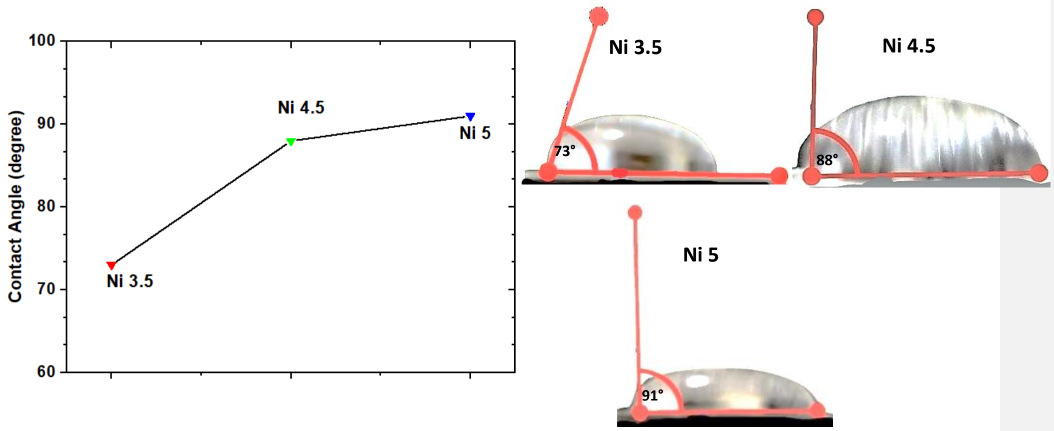


Figure 4: Wettability of the various Ni films deposited on the Al alloy substrates at different time.

2.4. CONCLUSION AND RECOMMENDATION

Nickel plating has been successfully carried out on the Al substrate with time variations. The effect of Ni coating affects surface morphology, crystal orientation, crystallite size and wettability. The Ni 4.5 sample has the largest crystal size of 44nm with the most dominant crystal plane [111] this is also confirmed by calculating the orientation index, this means that Ni elektropalting on aluminium with time variations has been successfully carried out. The morphological structure of the crystal is in the form of a pyramid completely distributed over the Al substrate as seen from the Gaussian plot. In the Ni 5 sample has hydrophobic properties with a contact angle of 91°. Even though the EDS characterization contained Ni oxide, but in XRD observations were not found because the O_2 that formed was very small

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EFFECT OF NICKEL ELECTROPLATING PROCESS TIME VARIATION OVER ALUMINUM ON CRYSTALPLANE ORIENTATION

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Abstract. Metal plating is starting to be widely used as a way to prevent metal corrosion. Apart from preventing corrosion, metal coatings were being developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. The purpose of this research is to make Ni over Al coating in time variation differences (3.5 h, 4.5 h and 5 h) with a constant current of 50 mA cm-

2. The characterization of the materials used was from SEM-EDS, XRD and digital cameras. The SEM characterization results showed a crystal shape similar to a pyramid and contained Ni oxide content in the Ni coating, although in the XRD observations did not appear. The increasing of the coating time will increase the crystal size in the direction of the preferred plane orientation

[111] which was confirmed by the calculation of the orientation index. The Ni 5 sample has hydrophobic properties with a contact angle of 91 °.

Keywords: electrodeposition,hydrophobic,
SEM

3.1. INTRODUCTION

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3.2. METHOD

The anode rod used is 98% pure nickel metal and the anode is an aluminum substrate. The electrolyte solution used was a watts solution of NiSO4.6H2O (210g L-1), NiCl2.6H2O (20 gL-1) and H3BO3 (45 g L-1) with quality / grade analyst from the chemical manufacturer Merck. Before the experiment, the aluminum metal was sanded and washed in an ultrasonic solution. The watts solution to be used is treated first by stirring for 1 hour with a magnetic stirred (Bante Instrument MS 3000) at a speed of 200 rpm to get homogeneity. The electroplating experiment used a current of 50 mA cm-2 with a time variation of 3.5 hours, 4.5 hours and 5 hours.

TABLE 5. Chemical composition of the Al alloy substrates (wt%), determined from the X-ray fluorescence measurement

Element	Al	Mg	Fe
Concentration	96,88	1,49	1,63

Each sample will be written as Ni 3.5, Ni 4.5 and Ni 5 to represent each experiment with time variation in this study. The anode rod used in this research has the following composition:

TABLE 6. Chemical composition of the pure Ni (wt%), determined from the X-ray fluorescence measurement.

Element	**Concentration (%)**
Al (Alumunium)	0,02
Ca (Kalsium)	0,04
Fe (Besi)	0,23
Ni (Nikel)	**98,01**
Y (Yitrium)	1,61
Zr (Zirkon)	0,04
Nb (Niobium)	0,05

Surface morphological analysis of the nickel plating experiment on aluminum using FE-SEM (FE-SEMFEI INSPECT F50 EDAX EDS Analyzer) with magnifications of 2500 × and 10000 ×. The crystalline structure of the Nickel layer was identified by XRD (Rigaku RINT 2000 with Cu K radiation). XRD data is processed with the help of HighScore Plus software to refined and calculate the crystal size, besides that, the position of the particles can also be known.

Water contact angle observations were taken using a Canon 1000D EOS camera. The value determines criteria of the angle θ as $\theta < 90°$, $90° \leq \theta < 150°$, $150° \leq \theta < 180°$ for being hydrophilic, hydrophobic, and superhydrophobic, respectively [28]

3.3. RESULT AND DISCUSSION

Electroplating is used to form a microstructural pattern of nickel plating over aluminum. The time variation will provide information about changes in the shape of the surface morphology and affect the size of the crystals(3.5 h, 4.5 and 5). In addition, we can determine the crystal size distribution using statistical analysis with ImageJ (Oppen Access) and Origin (OriginLab Corporation). By measuring the length of the grain diameter by ImageJ, then processing it by Origin.

Figure 1 shows the microstructure evolution of pure Nickel plating over aluminum with time variations. It can be seen from the SEM results that Nickel is evenly distributed over the Aluminum substrate in the electroplating process with a time of 3.5 h (**Figure 1a**). Then it can be seen visually from the increase in time to 4.5 h the crystal size is getting bigger and looks quite close to each other (**Figure 1b**). A customarily distributed Gaussian pattern can confirm this. Finally, at the time variation of 5 h, the crystals became visually more compact and bonded to one another (**Figure 1c**).

As additional information, it can be seen that all layers of Ni 3.5 - Ni 5 (**Figure. 1 a - c**), showed a half pyramidal structure with a grain area (8 - 30 µm) [30]. This pyramidal structure is morphologically typical for Ni plating with a preferred plane texture in a plane. [100,111] [31] - [33].

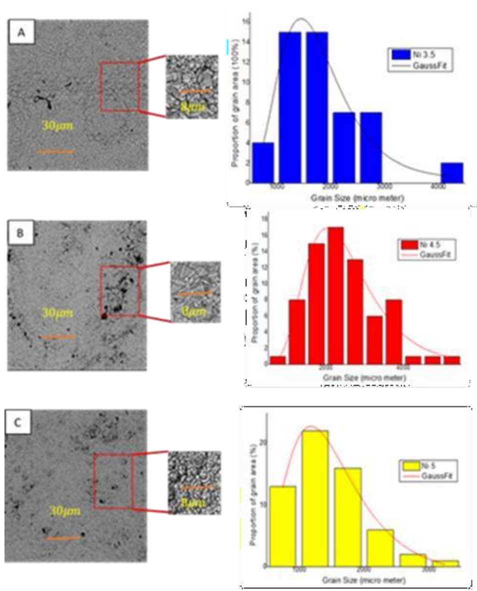

FIGURE 5 (a-c). SEM micrographs for different layers show the morphology of pure Ni coating separately deposited and distribution particle obtained using ImageJ combine Origin with corresponding Gauss fitting function

In the electroplating process, an anodizing event occurs. The cathode attracts the positive ions because a unidirectional electric current flows between the two electrodes in the electrolyte solution. Apart from releasing positive ions, the anode electrode also releases oxygen. Oxygen gas needs to be considered because as an inhibitor for anode electrode to releases positive ions [34], [35].

The presence of oxygen gas can be seen with the EDS pattern of nickel electroplating on an aluminum substrate presented in **Figure 2**. EDS analyzes the presence of significant peaks of nickel and oxygen peaks.

Transferring positive ions from the anode to the cathode causes a small amount of carbon to accumulate in the Ni anode rod. Readings of how much the weight percent Ni, C and O2 can be seen in table 3.

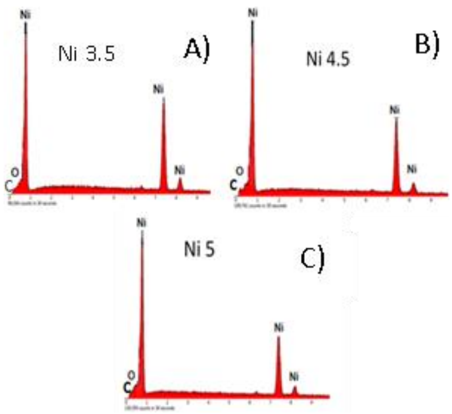

FIGURE 6. EDS pattern showing an element of Ni, C and O at various times

TABLE 3. Ni, C and O content on electrodeposit Ni Film with different time

Sampel	Ni (wt%)	C (wt%)	O (wt%)
Ni 3.5	98.30	0.90	0.80
Ni 4.5	96.86	2.00	1.14
Ni 5	95.47	2.41	2.12

When viewed from the data in table 3, it can be seen that the Ni 3.5 sample weight percent Ni 98.30 (wt%),C 0.90 (wt%) and O2 0.80 (wt%). Then for the sample Ni 5 has a weight percent Ni 95.47 (wt%), C 2.00 (wt%) and O2 1.14 (wt%). It can conclud that the increasing variety of time will increase the weight percent (wt%) of O2. the increase in O2 comes from oxidized Ni, but even though EDS detected O2, it was not observed by XRD

The calculation of the µ orientation index aims to determine the crystal structure growing or even shrinking along with the electroplating process with time variations. The orientation index µ (hkl) is calculated using the equation:

$$\mu\,(hkl) = \frac{I(hkl)/I_0(hkl)}{\frac{1}{n}\sum I(h^F k^F l^F)/I_0\,(h^F k^F l^F)}$$

Where is the peak intensity of each crystal plane from the research data, while is the peak intensity of the crystalis obtained from the ICSD (Inorganic Crystal Structure Database). In addition, $I(h'k'l')$ is the summation of the three peak intensities from the research data and $I_0(h'k'l')$ is the summation of the three peak intensities from the ICSD data. An index value greater than 1 $[\mu(hkl) \geq 1]$ indicates that the crystal field (hkl) is growing, meanwhile, if $[\mu(hkl) \leq 1]$ indicates that the crystal field is shrinking.

TABLE 4. Calculation of automatic index for coating Ni over Al with time variation

Sample	hkl			μ(hkl)		
	[111]	[002]	[022]	[111]	[002]	[022]
Ni 3.5	3702	1950	668	0,70	1,18	1,08
Ni 4.5	8570	967	619	1,63	0,58	1,00
Ni 5	3466	2006	557	0,66	1,22	0,90

3.3.1. In this study, the Ni over Al coating with the current density was kept constant at 50 mA cm^{-2} with timevariations showing that the crystal plane grew and shrank from the calculation of the orientation index μ.In the sample Ni 4.5, the crystal plane [111] known to have a value of 1.63, which means that compared to Ni 3.5, the crystal plane grows more due to the addition of time. Crystal field [022] experienced growth in the Ni 3.5 and Ni 5 samples, but shrank in the Ni5 samples. This is due to a decrease in electrical conductivity from the use of solution concentration..

The time variation effect affects the growth orientation of a particular crystal plane. The longer the plating time will affect the orientation of a certain crystal plane. This is estimated from the higher concentration of Ni^{2+} ions in solution. The growth of plane orientation [111] has advantages including anti-corrosion, increasing electrical conductivity and increasing mechanical properties [36].

3.3.2. The results of the plane orientation in table 4 are confirmed by the XRD pattern produced. The XRD pattern is shown in Figure 3 results from the linear refinement of the XRD pattern processed using the HighscorePlus Software. The crystal structure formed is a face centered cubic (FCC) which shows three significant peaks for the plane [111], [002] and [022]. The phase formed is a single phase and no other phases are formed on the surface of the Ni layer. The results of XRD data processing did not show nickel oxide, even though O_2 was detected by EDS (Figure 2), because the oxide formed in Ni was small.

There are three significantly different peak intensities, the peaks are the plane [111], [002] and[022]. The difference in peak intensity is due to the difference in mass density on the surface of each crystal plane due to time variations.

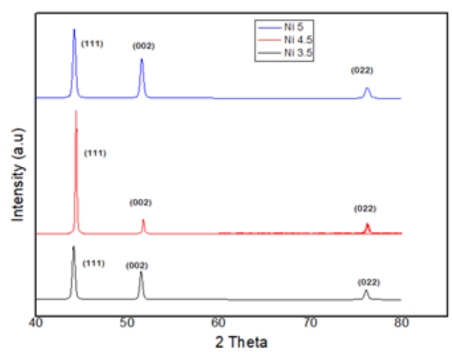

FIGURE 3. XRD pattern of electrodeposit Ni onto Al substrate with various time at constant current densities 50mA cm^{-2}

3.3.3. **The calculation of crystal size uses the Debye-Scherrer equation, where k is a constant of 0.9, β (the width of the diffraction peak of FWHM in radians, λ (the x-ray wavelength using Cu Kα radiation is 0.154nm), and θ (Bragg's angle) [37].**

$$D = \frac{k\lambda}{\beta \cos \theta}$$

3.3.4. **The crystal parameters of the sample after doing Rietvel Refinement using HighScore Plus software arepresented in table 5. Rietveld corrections were made until the GOF (Goodness of fit) value was less than2.5% and the ROP (R-weighted pattern) value was less than 10%. The atom's position can also be seen in the table which states that the atom's position occupies every point in the cube with its occupancy value of 1, so it can be concluded that there is no atomic shift.**

TABLE 5. Crystal parameter of Ni films at various times and constant current densities at 50mA cm^{-2}

Parameter		**Sample**		
		Ni 3.5	**Ni 4.5**	**Ni 5**
Crystal structure		Cubic FCC	Cubic FCC	Cubic FCC
Space Group		Fm-3m	Fm-3m	Fm-3m
space Group Number		225	225	225
Laticce constan (Å) a = b = c		3,5175	3,5206	3,5152
Volume (Å$_3$)		44.52	43,64	43,44
Densitas (g/cm3)		8,79	8,88	8,79
d-spacing (Å)		1,0142	1,0093	2,04906
Crystallite Size Debye-Scherrer (nm)		24	42	23
Atomic Position	X	0	0	0
	Y	0	0	0
	Z	0	0	0
	Occupancy	1	1	1
R-weighted pattern		7,67075	9,09029	7,249
GOF (Goodness of Fit)		0,74151	1,00618	1,986

Determination of hydrophilic and hydrophobic properties can be done using a water drop test over the sample. **Figure 4** presents a photo of the results of the drop test that has been carried out. In the drop test, it can be seen that there is a difference in contact angle due to time variations. Therefore, it can be concluded that over time the angle of contact increases as well. This has also been observed by [38] in their study. Samples Ni 3.5 and Ni 4.5 show hydrophilic properties because the contact angle is 73 ° and 88 °. Ni 5 sample has hydrophobic properties because it has a contactangle of 91 ° where the range for hydrophobic properties (90 ° ≤ θ <150 °) [38].

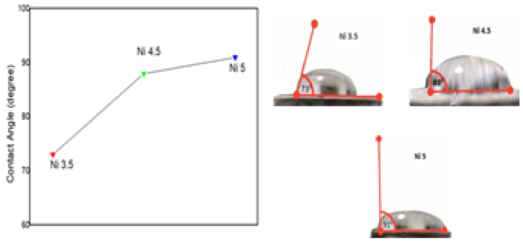

Figure 4: Wettability of the various Ni films deposited on the Al alloy substrates at different time..

3.4. CONCLUSION AND RECOMMENDATION

Nickel plating has been successfully carried out on the Al substrate with time variations. The effect of Ni coating affects surface morphology, crystal orientation, crystallite size and wettability. The Ni 4.5 sample has the largest crystal size of 44nm with the most dominant crystal plane [111] this is also confirmed by calculating the orientation index. The morphological structure of the crystal is in the form of a pyramid completely distributed over the Al substrate as seen from the Gaussian plot. In the Ni 5 sample has hydrophobic properties with a contact angle of91 °. Even though the EDS characterization contained Ni oxide, but in XRD observations were not found because theO2 that formed was very small

3.5. REFERENCES

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4. Keputusan Editor
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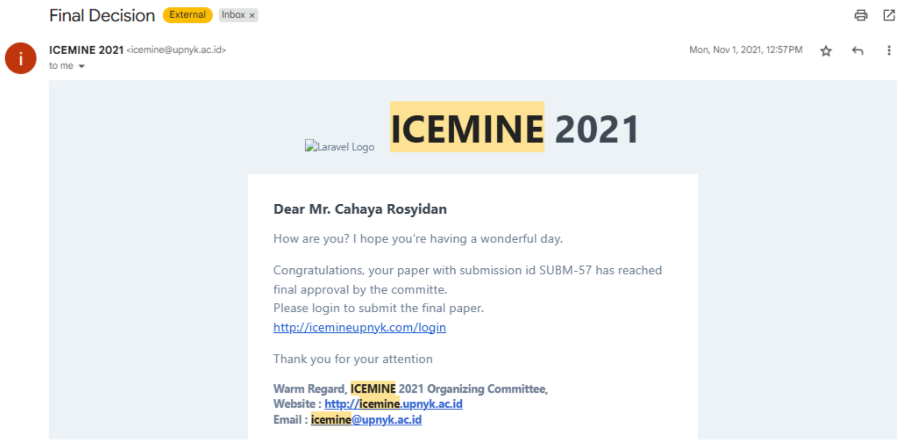
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5. Bukti Artikel Terbit
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RESEARCH ARTICLE | JUNE 12 2023

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AIP Conference Proceedings 2598, 040009 (2023)
https://doi.org/10.1063/5.0126191

Effect Of Nickel Electroplating Process Time Variation Over Aluminum On CrystalPlane Orientation

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Abstract. Metal plating starts to be widely used as a way to prevent metal corrosion. Apart from preventing corrosion, metal coatings were being developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. The purpose of this research is to make Ni coating over Al substrate in time variation differences (3.5 h, 4.5 h and 5 h) with a constant current of 50 mA cm-2. The characterization of the materials used was from SEM-EDS, XRD and digital cameras. The SEM characterization results showed a crystal shape similar to a pyramid and contained Ni oxide content in the Ni coating, although in the XRD observations did not appear. The increasing of the coating time will increase the crystal size in the direction of the preferred plane orientation
[111] which was confirmed by the calculation of the orientation index. The Ni 5 sample has hydrophobic properties with a contact angle of 91 °.

Keywords: electrodeposition,hydrophobic,SEM

INTRODUCTION

Aluminum metal is one of the materials that is widely used by people such as transportation, buildings and construction as well as home accessories and decorations because of its soft and stylish appearance, its color is silvery and shiny. Prices are quite affordable and easy to obtain, different uses of aluminum metal in the engineering field The use of aluminum metal in the engineering field is easy to form, a good heat conductor, resistant to cold temperatures, and aluminum can also be corroded. [1], [2]

Metal plating is starting to be widely used as a way to prevent metal from corrosion. In addition to preventing corrosion, metal coatings have been developed to thicken the metal, increase its hardness, wear resistance and corrosion resistance. [3] - [6]. Many factors affect to get this benefits results which are the composition of the material, the composition of the solution [7], [8], the temperature of the solution [9] - [12], the current density of the solution [13] - [15], the duration of immersion and the electric voltage [5], [13] - [20]. The electroplatingprocess has long been recognized for plating metals such as nickel, chrome, gold and silver. Electroplating method offers many advantages such as faster, reliable and relatively cheaper [21] [22] [23] - [26].

The crystal size has a direct impression on the surface energy of the coating. Along with surface energy, surface morphology also plays a vital role in surface wettability. Because of the change in current density (j), thickness,layer morphology and crystal size can be obtained. Therefore, this research will look for changes in the microstructureof the layer and the distribution of Ni particles on the Al substrate with time variations [27], [28].

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4th International Conference on Earth Science, Mineral and Energy
AIP Conf. Proc. 2598, 040009-1–040009-8; https://doi.org/10.1063/5.0126191
Published by AIP Publishing. 978-0-7354-4543-7/$30.00

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Zr (Zirkon)	0,04
Nb (Niobium)	0,05

Surface morphological analysis of the nickel plating experiment on aluminum using FE-SEM (FE-SEMFEI INSPECT F50 EDAX EDS Analyzer) with magnifications of 2500 × and 10000 ×. The crystalline structure of the Nickel layer was identified by XRD (Rigaku RINT 2000 with Cu K radiation). XRD data is processed with the help of HighScore Plus software to refined and calculate the crystal size, besides that, the position of the particles can also be known.

Water contact angle observations were taken using a Canon 1000D EOS camera. The value determines criteria of the angle θ as $\theta < 90°$, $90° \leq \theta < 150°$, $150° \leq \theta < 180°$ for being hydrophilic, hydrophobic, and superhydrophobic, respectively [28]

RESULT AND DISCUSSION

Electroplating is used to form a microstructural pattern of nickel plating over aluminum. The time variation will provide information about changes in the shape of the surface morphology and affect the size of the crystals(3.5 h, 4.5 and 5). In addition, we can determine the crystal size distribution using statistical analysis with ImageJ (Oppen Access) and Origin (OriginLab Corporation). By measuring the length of the grain diameter by ImageJ, then processing it by Origin.

Figure 1 shows the microstructure evolution of pure Nickel plating over aluminum with time variations. It can be seen from the SEM results that Nickel is evenly distributed over the Aluminum substrate in the electroplating process with a time of 3.5 h (**Figure 1a**). Then it can be seen visually from the increase in time to 4.5 h the crystal size is getting bigger and looks quite close to each other (**Figure 1b**). A customarily distributed Gaussian pattern can confirm this. Finally, at the time variation of 5 h, the crystals became visually more compact and bonded to one another (**Figure 1c**).

As additional information, it can be seen that all layers of Ni 3.5 - Ni 5 (**Figure. 1 a - c**), showed a half pyramidal structure with a crystal (8 - 30 µm) [30]. This pyramidal structure is morphologically typical for Ni plating with apreferred plane texture in a plane. [100,111] [31] - [33].

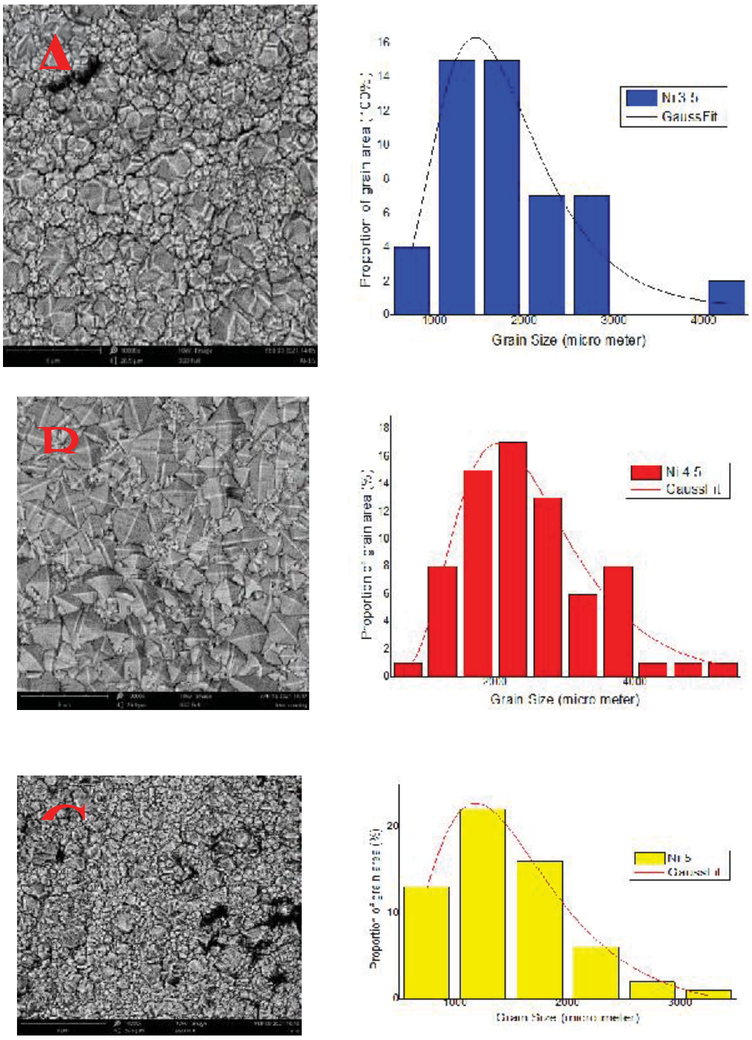

FIGURE 1 (a-c). SEM micrographs for different layers show the morphology of pure Ni coating separately deposited and distribution particle obtained using ImageJ combine Origin with corresponding Gauss fitting function

In the electroplating process, an anodizing event occurs. The cathode attracts the positive ions because a unidirectional electric current flows between the two electrodes in the electrolyte solution. Apart from releasing positive ions, the anode electrode also releases oxygen. Oxygen gas needs to be considered because as an inhibitor for anode electrode to releases positive ions [34], [35].

The presence of oxygen gas can be seen with the EDS pattern of nickel electroplating on an aluminum substrate presented in **Figure 2**. EDS analyzes the presence of significant peaks of nickel and oxygen peaks.

Transferring positive ions from the anode to the cathode causes a small amount of carbon to accumulate in the Ni anode rod. Readings of how much the weight percent Ni, C and O2 can be seen in table 3.

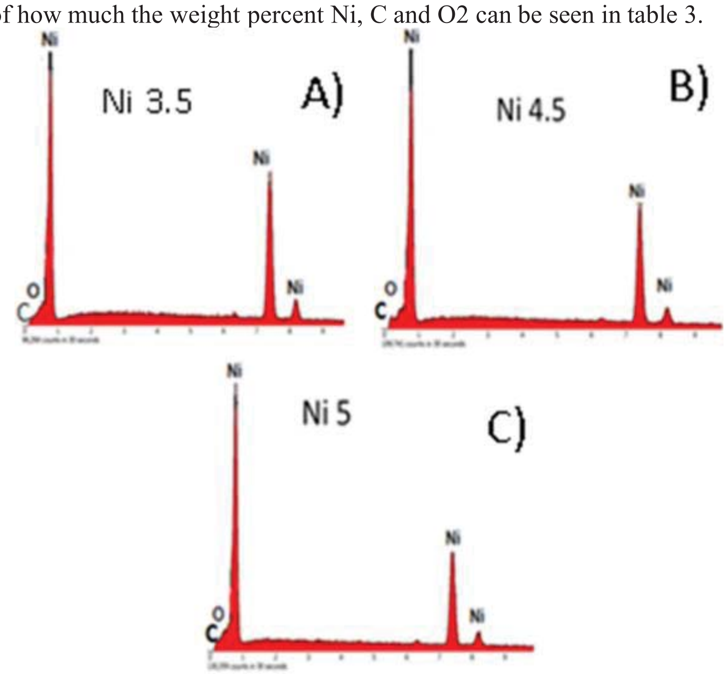

FIGURE 2. EDS pattern showing an element of Ni, C and O at various times

TABLE 3. Ni, C and O content on electrodeposit Ni Film with different time

Sampel	Ni (wt%)	C (wt%)	O (wt%)
Ni 3.5	98.30	0.90	0.80
Ni 4.5	96.86	2.00	1.14
Ni 5	95.47	2.41	2.12

When viewed from the data in table 3, it can be seen that the Ni 3.5 sample weight percent Ni 98.30 (wt%),C 0.90 (wt%) and O2 0.80 (wt%). Then for the sample Ni 5 has a weight percent Ni 95.47 (wt%), C 2.00 (wt%) and O2 1.14 (wt%). It can conclud that the increasing variety of time will increase the weight percent (wt%) of O2. the increase in O2 comes from oxidized Ni, but even though EDS detected O2, it was not observed by XRD

The calculation of the μ orientation index aims to determine the crystal structure growing or even shrinking along with the electroplating process with time variations. The orientation index μ (hkl) is calculated using the equation:

$$\mu(hkl) = \frac{I(hkl)/I_0(hkl)}{\frac{1}{n}\sum I(h^F k^F l^F)/I_0(h^F k^F l^F)}$$

Where is the peak intensity of each crystal plane from the research data, while is the peak intensity of the crystalis obtained from the ICSD (Inorganic Crystal Structure Database). In addition, $I(h'k'l')$ is the summation of the three peak intensities from the research data and $I_0(h'k'l')$ is the summation of the three peak intensities from the ICSD data. An index value greater than 1 [$\mu(hkl) \geq 1$] indicates that the crystal field (*hkl*) is growing, meanwhile, if [$\mu(hkl) \leq 1$] indicates that the crystal field is shrinking.

TABLE 4. Calculation of automatic index for coating Ni over Al with time variation

Sample	hkl			μ(hkl)		
	[111]	[002]	[022]	[111]	[002]	[022]
Ni 3.5	3702	1950	668	0,70	1,18	1,08
Ni 4.5	8570	967	619	1,63	0,58	1,00
Ni 5	3466	2006	557	0,66	1,22	0,90

In this study, the Ni over Al coating with the current density was kept constant at 50 mA cm^{-2} with timevariations showing that the crystal plane grew and shrank from the calculation of the orientation index μ.In the sample Ni 4.5, the crystal plane [111] known to have a value of 1.63, which means that compared to Ni 3.5, the crystal plane grows more due to the addition of time. Crystal field [022] experienced growth in the Ni 3.5 and Ni 5 samples, but shrank in the Ni5 samples. This is due to a decrease in electrical conductivity from the use of solution concentration..

The time variation effect affects the growth orientation of a particular crystal plane. The longer the plating time will affect the orientation of a certain crystal plane. This is estimated from the higher concentration of Ni^{2+} ions in solution. The growth of plane orientation [111] has advantages including anti-corrosion, increasing electrical conductivity and increasing mechanical properties [36].

The results of the plane orientation in table 4 are confirmed by the XRD pattern produced. The XRD pattern is shown in **Figure 3** results from the linear refinement of the XRD pattern processed using the HighscorePlus Software. The crystal structure formed is a face centered cubic (FCC) which shows three significant peaks for the plane [111], [002] and [022]. The phase formed is a single phase and no other phases are formed on the surface of the Ni layer. The results of XRD data processing did not show nickel oxide, even though O_2 was detected by EDS (**Figure 2**), because the oxide formed in Ni was small.

There are three significantly different peak intensities, the peaks are the plane [111], [002] and[022]. The difference in peak intensity is due to the difference in mass density on the surface of each crystal plane due to time variations.

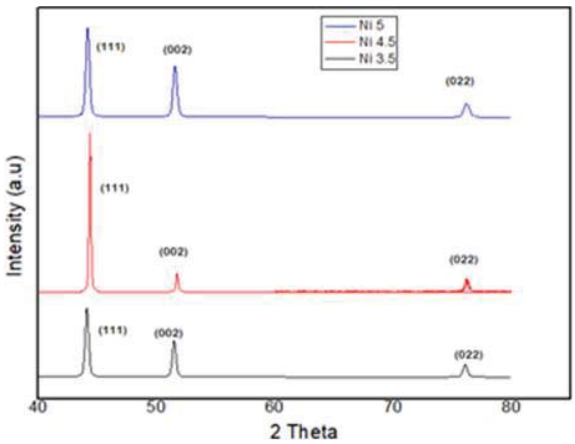

FIGURE 3. XRD pattern of electrodeposit Ni onto Al substrate with various time at constant current densities 50mA cm^{-2}

The calculation of crystal size uses the Debye-Scherrer equation, where k is a constant of 0.9, β (the width of the diffraction peak of FWHM in radians, λ (the x-ray wavelength using Cu Kα radiation is 0.154nm), and θ (Bragg's angle) [37].

$$D = \frac{k}{\beta \cos \theta}$$

The crystal parameters of the sample after doing Rietvel Refinement using HighScore Plus software arepresented in table 5. Rietveld corrections were made until the GOF (Goodness of fit) value was less than2.5% and the ROP

(R-weighted pattern) value was less than 10%. The atom's position can also be seen in the table which states that the atom's position occupies every point in the cube with its occupancy value of 1, so it can be concluded that there is no atomic shift.

TABLE 5. Crystal parameter of Ni films at various times and constant current densities at 50mA cm^{-2}

Parameter		Sample Ni 3.5	Ni 4.5	Ni 5
Crystal structure		Cubic FCC	Cubic FCC	Cubic FCC
Space Group		Fm-3m	Fm-3m	Fm-3m
space Group Number		225	225	225
Laticce constan (Å)a = b = c		3,5175	3,5206	3,5152
Volume ($Å_3$)		44.52	43,64	43,44
Densitas (g/cm3)		8,79	8,88	8,79
d-spacing (Å)		1,0142	1,0093	2,04906
Crystallite Size Debye-Scherrer(nm)		24	42	23
Atomic Position	X	0	0	0
	Y	0	0	0
	Z	0	0	0
	Occupancy	1	1	1
R-weighted pattern		7,67075	9,09029	7,249
GOF (Goodness of Fit)		0,74151	1,00618	1,986

Determination of hydrophilic and hydrophobic properties can be done using a water drop test over the sample. **Figure 4** presents a photo of the results of the drop test that has been carried out. In the drop test, it can be seen that there is a difference in contact angle due to time variations. Therefore, it can be concluded that over time the angle of contact increases as well. This has also been observed by [38] in their study. Samples Ni 3.5 and Ni 4.5 show hydrophilic properties because the contact angle is 73 ° and 88 °. Ni 5 sample has hydrophobic properties because it has a contactangle of 91 ° where the range for hydrophobic properties (90 ° ≤ θ <150 °) [38].

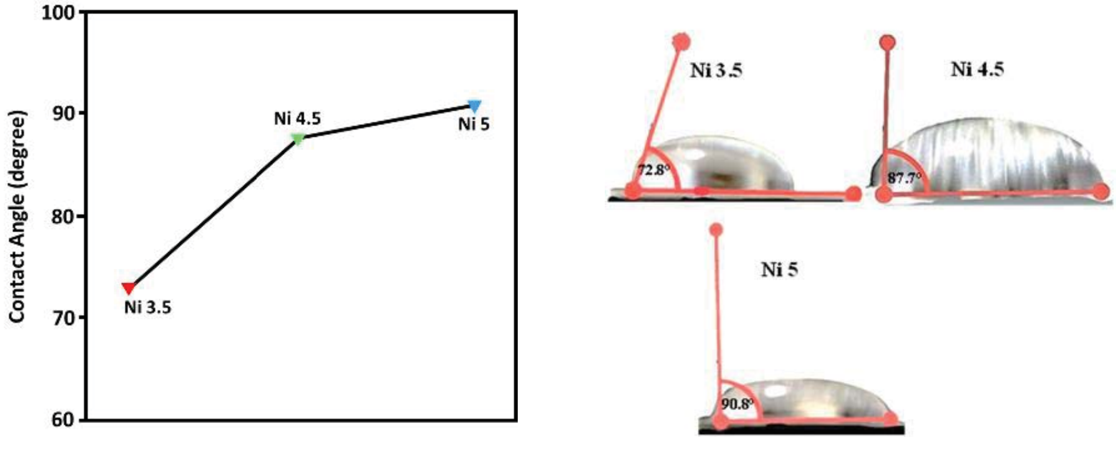

Figure 4: Wettability of the various Ni films deposited on the Al alloy substrates at different time.

CONCLUSION AND RECOMMENDATION

Nickel plating has been successfully carried out on the Al substrate with time variations. The effect of Ni coating affects surface morphology, crystal orientation, crystallite size and wettability. The Ni 4.5 sample has the largest crystal size of 44nm with the most dominant crystal plane [111] this is also confirmed by calculating the orientation index, this means that Ni elektropalting on aluminium with time variations has been successfully carried out. The morphological structure of the crystal is in the form of a pyramid completely distributed over the Al substrate as seen from the Gaussian plot. In the Ni 5 sample has hydrophobic properties with a contact angle of91 °. Even though the EDS characterization contained Ni oxide, but in XRD observations were not found because theO2 that formed was very small

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