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# Determining the Particle Size of Cu and Ni in Thin Cu/Ni Films using the Williamson-Hall Method

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***Abstract*—This research focuses on analyzing the particle size of a thin Cu/Ni layer produced through electroplating by varying the input voltage. The Williamson-Hall method is used to determine the particle size of the layer, and an X-ray diffractometer is used to characterize the layer. The study finds that the particle size of Cu and Ni layers with different applied voltages has different values due to various factors. The optimum voltage for the Ni layer is found to be 7.5 volts, and its overall particle size is 4.13 × 10^(-10) nm, while the particle size of Cu is 5.00 × 10^(-9) nm. The applied voltage affects the particle size produced, and the research identifies an optimum voltage at 7.5 volts.**

***Index Terms*—Particle Size, Thin Films, Williamson-Hall Method**

## I. Introduction

ENGINEERING technology development in the 21st century has brought many advancements to various aspects of life. It based on Nanomaterials enables the creation of new materials with specifications tailored to current needs [1]. Engineering can be added by modifying the structure, adding composition, or forming certain phases. Currently, much-developed research is focused on materials with Nano crystal sizes [2].

Nature is created with the diversity of materials existing within it, from materials in the form of gases and liquids to solids. Humans interact with all three materials, especially solid materials with Nano sizes, which have become the focus of human engineering in advancing civilization. Solids or solid materials are atoms that occupy fixed positions, space, and mass.

Particle size is an essential parameter in electrolyte analysis to determine the resistivity value of a material [3]. Studies of the physics properties of a component material are considered important in supporting technological advances, especially in the form of thin films created through electroplating [4]. Electroplating is depositing a metal layer on an electrode that aims to form a surface with different properties and dimensions (thickness) than the base metal [5]. For example, creating a thin nickel layer that coats a copper sheet by giving variations in the voltage. After the procedure, by providing variations in voltage to the electrode, the rate of a metal ion as it moves towards the cathode will also vary (proportionally). It will affect the size of the deposited layer. Some of the materials made as coating and coating materials are Fe, Cu, Ni, Cr, and Li. However, in this research, we only use Cu and Ni samples.

In classifying material, initial information is needed about the form or structure of the material. If a material is composed of atoms with a regular pattern, the material can be called a crystal. If a material is composed of atoms that have distinctive ways, it can be called amorphous [6]. The particle is a crystal surrounded by other crystals with the same type but different orientations. If the grain size of a material is large, then the resulting resistivity value will be small. The resulting resistivity value will be considered if the grain size is small [7].

Some reading materials use several methods in particle size analysis, such as Scanning Electron Microscope (SEM), Image J, and X-Ray Diffraction (XRD) [8]. The most commonly used equation is the Scherrer equation and the Williamson-Hull plot [9]-[11]. The Scherrer equation to analyze the particle size of a material still has limitations. The Scherrer equation only considers particle size in the peak broadening using XRD but cannot provide other information about the microcrystalline structure, especially for complex materials.

The objectives of this research are:

1. To analyze the method of determining particle size in thin Cu/Ni layers using the Williamson-Hall plot.
2. To analyze the effect of voltage variation on particle size.

## II. Method

This research aims to determine and understand the particle size of a thin layer of Cu/Ni produced by electroplating by varying the input voltage, which is then analyzed using the Williamson-Hall method. The thin layer of Cu/Ni is characterized using an X-ray diffractometer using the working principle of X-ray diffraction (XRD) [8]. Then from this stage, data will be analysed using the Williamson-Hall equation, resulting in the particle size [9].

The sample used is a sterilized copper sheet coated with Nickel through electroplating, with voltage variation from 6.0, 6.5, 7.0, 7.5, and 8.0 volts. The method used to analyse the Cu/Ni layer's particle size is the Williamson-Hall plot. The data from the XRD was carried out in the Laboratory of Sensors and Transducers located Universitas Ahmad Dahlan. Furthermore,

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the study uses the same sample to see the effect of voltage variation on particle size.

*A. Sample preparation technique*

Sample preparation technique are as follow:

1. Cut the copper sheet to the desired size of 1 cm × 1 cm.
2. Clean the sample mechanically by sandblasting using a sandblaster of size 5000. Rinse the sample with distilled water to remove any residual dirt.
3. Clean the sample again using a small amount of aerosol with the help of a tissue, then rinse the sample with distilled water to remove any residual dirt.
4. Clean the sample with a UV cleaner given distilled water for 5 minutes.
5. Dry the sample using a hairdryer.
6. Check the sample's resistivity.
7. The sample is now ready to use.

*B. Data Analysis Techniques*

The following are the steps to analyze the data obtained to determine the particle size. Peak determination and FWHM: In a sample that has been characterized using XRD, particularly in the thin layer of Cu/Ni used, the data obtained is in the form of 2 Theta (2θ) and Intensity (I). Then from the data obtained, a peak matching analysis was performed using the Match!3 software [12]. Besides determining the diffraction peak of the crystal, we can also see the broadening of the half-diffraction peak curve or the FWHM, which then, with this value, will be entered into the Williamson-Hall equation to determine the particle size. Determining the intercept value through the intercept value determination which performed after obtaining the 2θ peak value and FWHM.

Particle size determination can be performed after obtaining the intercept value from the sample, which is then plotted using the WH plot using the straight line equation. y = ax + b, with the value of an indicating the value of 4ε, the value of b indicating the value of log(K) where K is a constant, and the x-axis representing the FWHM. The particle size can be calculated using the equation D = (4ε/FWHM^2)^1/2.

## III. Result and Discussion

The sample used in this study is a Cu/Ni sample resulting from electroplating with varying deposition voltages. The distance between the anode and cathode plates is about 4cm, with a nickel anode and a copper cathode. The solution used was NiCl_2 with a temperature of 60 ℃. The electroplating process shows that the higher the voltage given, the faster the Ni ion movement and the greater the amount of Ni ions. As a result, the higher voltage will cause Ni coating on Cu to occur more quickly. The research data obtained from the XRD characterization results were then analyzed by matching each peak in the data to determine peak ownership and FWHM using the Match!3 software. Afterwards, the sample was analyzed using the Williamson-Hall method to find the particle size value [10]. The following is the calculation of particle size on thin Cu/Ni layers using deposition voltages of 6V, 6.5V, 7V, 7.5V, and 8V.

*A. Cu/Ni coating 6 volt voltage variation*

For the first calculation, the sample used is a Cu/Ni layer with a deposition voltage of 6 volts, and here is the XRD characterization result that was then analyzed using the assistance of the Match!3 software to determine the 2 theta value and ownership of each peak.

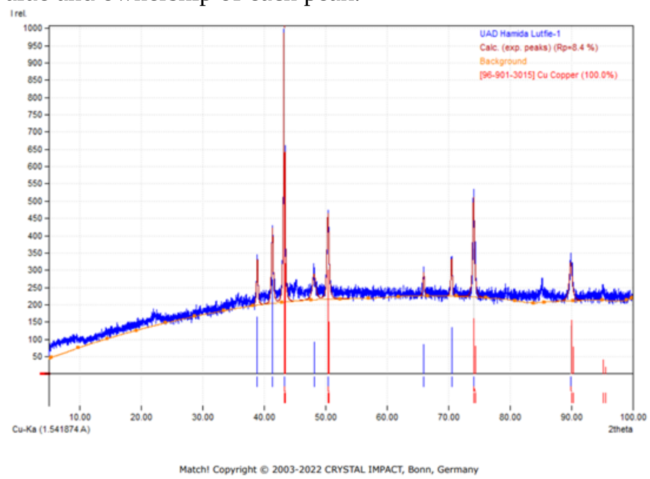


**Fig. 1.** XRD spectrum of Cu/Ni deposition voltage 6 volts

The FWHM and 2 theta values read by the Match!3 software will be presented in Table 1.

TABLE I
VALUE OF 2Θ AND FWHM CU/NI DEPOSITION VOLTAGE OF 6 VOLTS

| No. | $2\theta$ | FWHM |
|---|---|---|
| 1 | 38.94 | 0.2449 |
| 2 | 41.40 | 0.3051 |
| 3 | 43.32 | 0.2595 |
| 4 | 48.16 | 0.626 |
| 5 | 50.44 | 0.3736 |
| 6 | 66.00 | 0.2396 |
| 7 | 70.54 | 0.2549 |
| 8 | 74.12 | 0.3523 |
| 9 | 89.92 | 0.5568 |

Data in Table 1 is the data owned by the Cu/Ni sample, and Fig. 1 is an image obtained from the match!3 software. The red line is the data for the Cu layer, and the data with the blue line is the data for the Ni layer.

After obtaining the value of 2θ and the value of the FWHM on the Cu/Ni thin layer with a variation of the sample voltage of 6 volts, then look for the lattice strain value (ε), which can be obtained from the equation of the linear regression line divided by 4. The abscissa (x-axis) is sinθ, and the ordinate (y-axis) is β cosθ with the help of Microsoft Excel.

1) **Cu layer**

Fig. 2 displayed data regression analysis results.

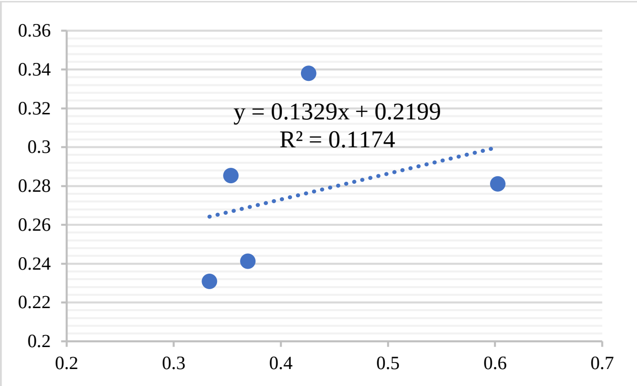


**Fig. 2.** Cu deposition voltage of 6 volts

Fig. 2 shows a graph of the linear equation resulting from the Cu layer at a deposition voltage of 6 volts. With the value obtained:

$y = 0.1329\,x + 0.2199$

The lattice strain value obtained from the 6 Volt Cu layer is 0.1329/4=0.033225 nm, so to determine the grain size it can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,2199 = \frac{0,9 \times (0,15406 \times 10^{-9})}{D}$$

$$0,2199D = 0,9 \times (0,15406 \times 10^{-9})$$

$$D = \frac{0,9 \times (0,15406 \times 10^{-9})}{0,2199} = 6,3053 \times 10^{-10}\, nm.$$

2) **Ni layer**

Fig. 3 displayed regressiondata analysis results.

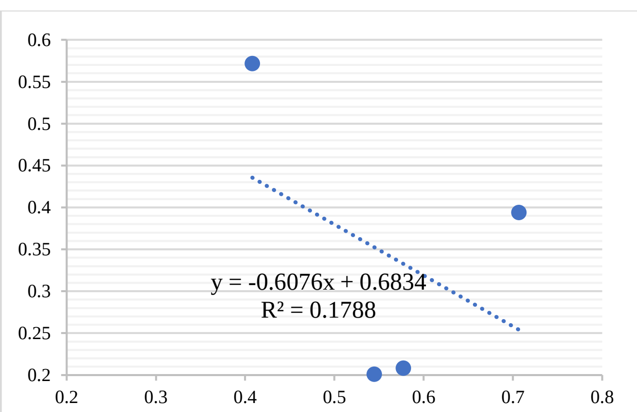


**Fig. 3.** Ni deposition voltage of 6 volts

Fig. 3 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 6 volts. With the value obtained:

$y = -0.6076\,x + 0.6834$

And for the lattice strain value of the 6 Volt Ni layer, it is (-0.6076)/4=-0.1519 nm, so to determine the grain size, it can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,6834 = \frac{0,9 \times (0,15406 \times 10^{-9})}{D}$$

$$0,6834D = 0,9 \times (0,15406 \times 10^{-9})$$

$$D = \frac{0,9 \times (0,15406 \times 10^{-9})}{0,6834} = 2,0289 \times 10^{-10}\, nm.$$

*B. Cu/Ni layer deposition voltage 6.5 volts*

For the second calculation, using a Cu/Ni layer sample with a given deposition voltage of 6.5 volts, the following is an image of the results of the XRD characterization, which is then analyzed using the help of Match!3 software to determine the value of 2θ and the ownership of each peak.

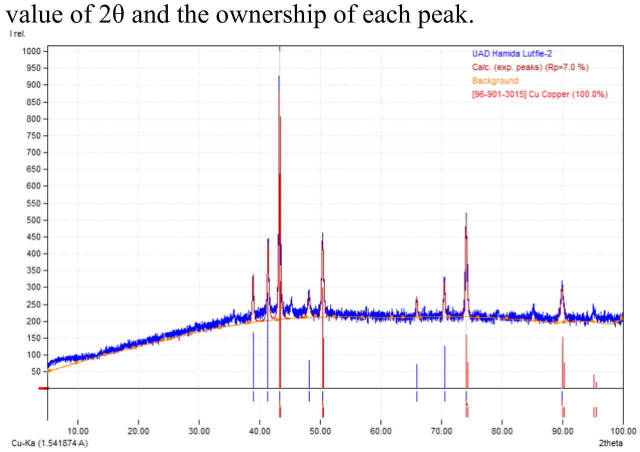


**Fig. 4.** XRD spectrum of Cu/Ni deposition voltage 6.5 volts

The FWHM and 2θ values that are read by the Match!3 software will be presented in Table II.

TABLE II
VALUE OF 2Θ AND FWHM CU/NI DEPOSITION VOLTAGE OF 6.5 VOLTS

| No. | 2θ | FWHM |
|---|---|---|
| 1 | 38.96 | 0.2803 |
| 2 | 41.44 | 0.2995 |
| 3 | 43.36 | 0.2616 |
| 4 | 48.20 | 0.4748 |
| 5 | 50.46 | 0.3559 |
| 6 | 66.00 | 0.228 |
| 7 | 70.56 | 0.2799 |
| 8 | 74.14 | 0.3386 |
| 9 | 89.94 | 0.5746 |

1) **Cu layer**

Fig. 5 shows the analysis results.

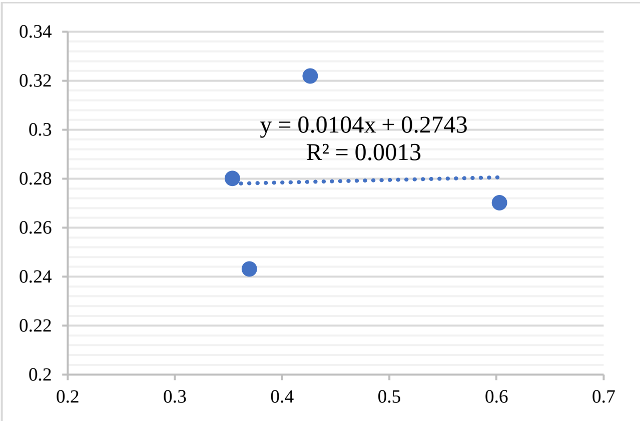


**Fig. 5.** Cu deposition voltage of 6.5 volts

Fig. 5 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 6 volts. with the value obtained

y = 0.0104 *x* + 0.2743

And for the lattice strain value of the 6 Volt Ni layer, it is 0.0104/4=0.0026 nm, so the grain size can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,2743 = \frac{0,9 \times (0,15406 \times 10^{-9})}{D}$$

$$0,2743D = 0,9 \times (0,15406 \times 10^{-9})$$

$$D = \frac{0,9 \times (0,15406 \times 10^{-9})}{0,2743} = 5,05483 \times 10^{-10}\,nm.$$

2) **Ni layer**

Fig. 6 shows the analysis results.

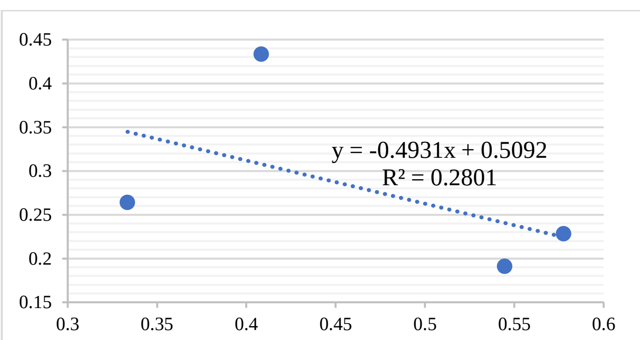


**Fig. 6.** Ni deposition voltage 6.5 volts

Fig. 6 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 6.5 volts. with the value obtained

*y* = -0.4931 *x* + 0.5092

And for the lattice strain value of the 6.5 Volt Ni layer, it is (-0.4931)/4=-0.123275 nm so that the grain size can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,5092 = \frac{0,9 \times (0,15406 \times 10^{-9})}{D}$$

$$0,5092D = 0,9 \times (0,15406 \times 10^{-9})$$

$$D = \frac{0,9 \times (0,15406 \times 10^{-9})}{0,5092} = 5,05483 \times 10^{-10}\,nm.$$

*C. Cu/Ni layer deposition voltage of 7 Volts*

For the third calculation, using a Cu/Ni layer sample with a given deposition voltage of 7 volts, the following is an image of the results of the XRD characterization, which is then analyzed using the help of Match!3 software to determine the value of 2θ and the ownership of each peak.

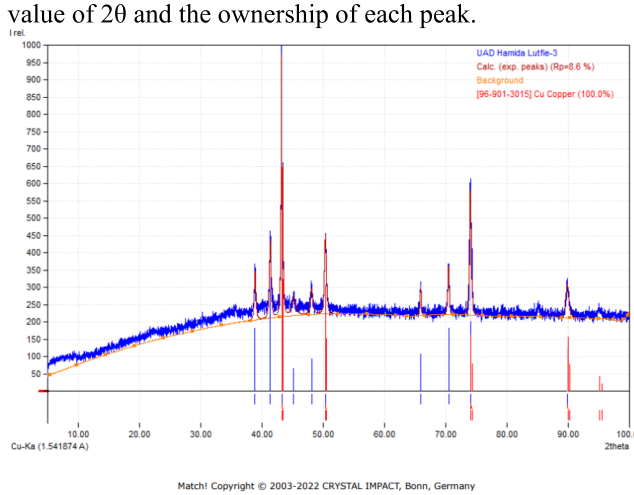


**Fig. 7.** XRD spectrum of Cu/Ni deposition voltage 7 volts

The FWHM and 2 θ values read by the Match!3 software will be presented in Table III.

TABLE III
VALUE OF 2Θ AND FWHM CU/NI DEPOSITION VOLTAGE OF 7 VOLTS

| No. | 2θ | FWHM |
|---|---|---|
| 1 | 38.94 | 0.3072 |
| 2 | 41.42 | 0.3249 |
| 3 | 43.32 | 0.2672 |
| 4 | 45.26 | 0.8082 |
| 5 | 48.16 | 0.4968 |
| 6 | 50.44 | 0.3576 |
| 7 | 66.00 | 0.2490 |
| 8 | 70.54 | 0.2706 |
| 9 | 74.1 | 0.3440 |
| 10 | 89.9 | 0.5578 |

1) **Cu layer**

Fig. 8 shows the analysis results to determine the value.

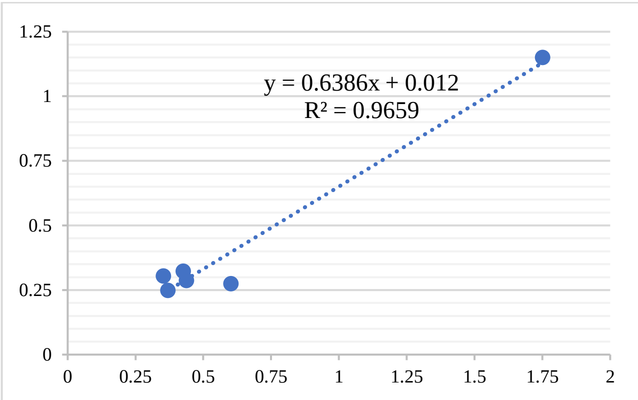


**Fig. 8.** Cu deposition voltage of 7 volts

Fig. 8 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 7 volts. with the value obtained

$y$ = 0.6386 $x$ + 0.012

And for the lattice strain value of the 7 Volt Cu layer is 0.6386/4=0.15965 nm, so the grain size can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,012 = \frac{0,9\times(0,15406\times10^{-9})}{D}$$

$$0,012D = 0,9\times(0,15406\times10^{-9})$$

$$D = \frac{0,9\times(0,15406\times10^{-9})}{0,012} = 1,1555\times10^{-8}\,nm.$$

2) **Ni layer**

Fig. 9 shows the analysis results.

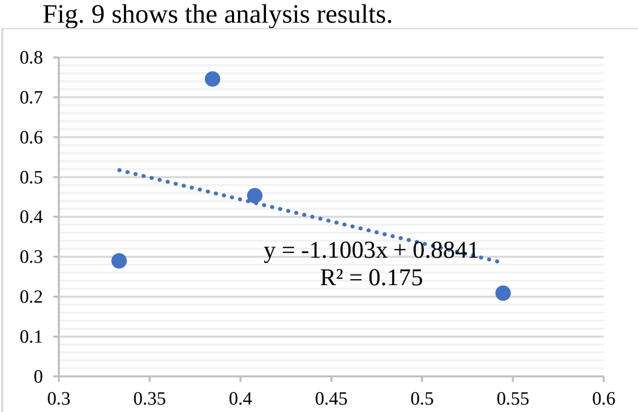


**Fig. 9.** Ni deposition voltage 7 volts

Fig. 9 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 7 volts. With the value obtained:

$y$ = -1.1003 $x$ + 0.8841

And for the lattice strain value of the 6 Volt Ni layer, it is (-1.1003)/4=0.275075 nm so that the grain size can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,8841 = \frac{0,9\times(0,15406\times10^{-9})}{D}$$

$$0,8841D = 0,9\times(0,15406\times10^{-9})$$

$$D = \frac{0,9\times(0,15406\times10^{-9})}{0,8841} = 1,5683\times10^{-10}\,nm.$$

*D. Cu/Ni layer deposition voltage 7.5 volts*

For the fourth calculation, using a Cu/Ni layer sample with a given deposition voltage of 7.5 volts, the following is an image of the results of the XRD characterization, which is then analyzed using the help of Match!3 software to determine the value of 2θ and the ownership of each peak.

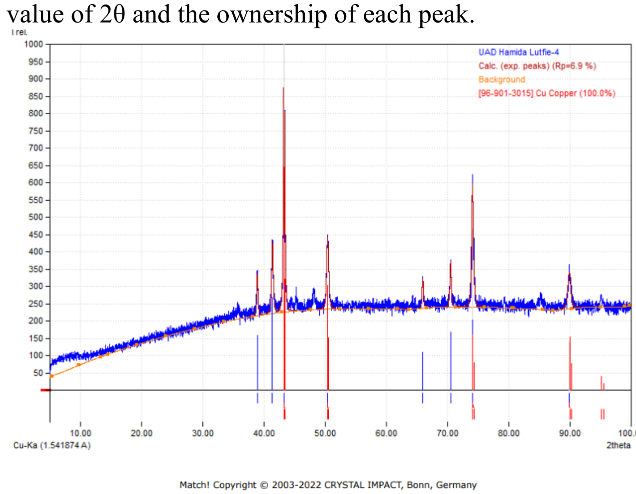


**Fig. 10.** XRD spectrum of Cu/Ni deposition voltage 7.5 volts

The FWHM and 2 θ values read by the Match!3 software will be presented in Table IV.

TABLE IV
VALUE OF 2Θ AND FWHM CU/NI DEPOSITION VOLTAGE OF 7.5 VOLTS

| No. | 2θ | FWHM |
|---|---|---|
| 1 | 38.98 | 0.2555 |
| 2 | 41.44 | 0.2976 |
| 3 | 43.36 | 0.2712 |
| 4 | 50.48 | 0.3615 |
| 5 | 66.02 | 0.2622 |
| 6 | 70.56 | 0.2697 |
| 7 | 74.14 | 0.3523 |
| 8 | 89.94 | 0.5532 |

1) **Cu layer**

Fig. 11 shows the analysis results to determine the value.

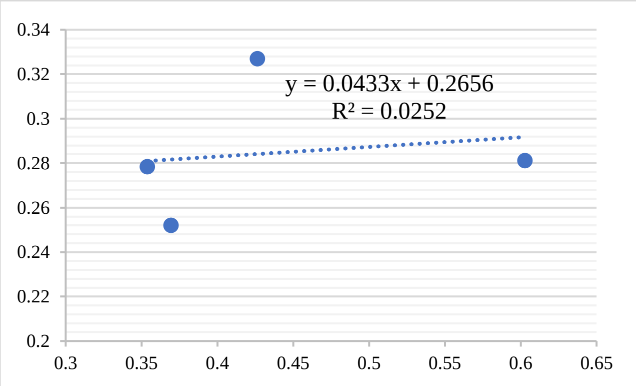


**Fig. 11.** Cu deposition voltage of 7.5 volts

Fig. 11 shows a graph of the linear equation resulting from the Cu layer at a deposition voltage of 7.5 volts. with the value obtained

$y = 0.0433\,x + 0.2656$

And for the lattice strain value of the 7.5 volt Cu layer, it is 0.0433/4=0.010825 nm, so the grain size can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,2656 = \frac{0,9\times(0,15406\times10^{-9})}{D}$$

$$0,2656D = 0,9\times(0,15406\times10^{-9})$$

$$D = \frac{0,9\times(0,15406\times10^{-9})}{0,2656} = 5,22041\times10^{-10}\,nm.$$

2) **Ni layer**

Fig. 12 shows the analysis results.

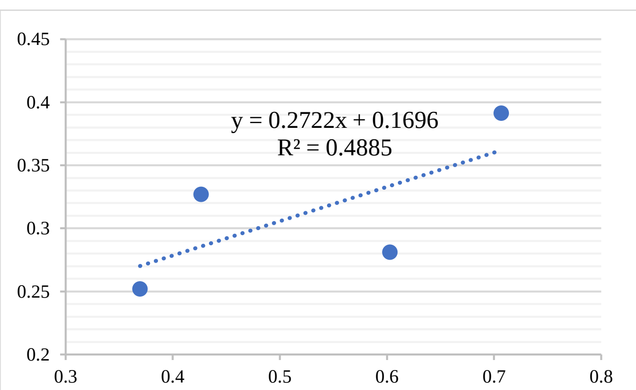


**Fig. 12.** Ni deposition voltage 7.5 volts

Fig. 12 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 7.5 volts. with the value obtained

$y = 0.2722\,x + 0.1696$

The lattice strain value of the 7.5 volt Ni layer is 0.27222/4=0.06805 nm, so to determine the grain size of the particles can be calculated using the equation:

$$b = \frac{K\lambda}{D}$$

$$0,1696 = \frac{0,9\times(0,15406\times10^{-9})}{D}$$

$$0,1696D = 0,9\times(0,15406\times10^{-9})$$

$$D = \frac{0,9\times(0,15406\times10^{-9})}{0,1696} = 8,17535\times10^{-10}\,nm.$$

*E. Cu/Ni thin film deposition voltage of 8 Volts*

For the fifth calculation, using a Cu/Ni layer sample with a given deposition voltage of 8 volts, the following is an image of the results of the XRD characterization, which is then analyzed using the help of Match!3 software to determine the value of 2θ and the ownership of each peak.

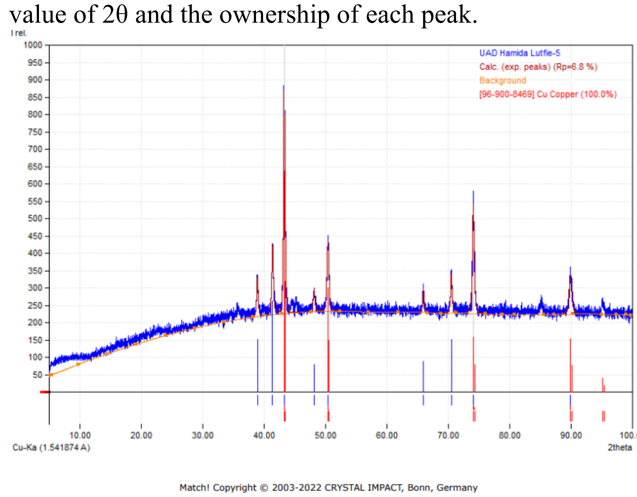


**Fig. 13.** XRD spectrum of Cu/Ni deposition voltage 8 volts

The FWHM and 2θ values read by the Match!3 software will be presented in the Table V.

TABLE V
THIS IS A SAMPLE OF A TABLE TITLE VALUE OF 2Θ AND FWHM CU/NI DEPOSITION VOLTAGE OF 8 VOLTS

| No. | 2θ | FWHM |
|---|---|---|
| 1 | 38.98 | 0.2626 |
| 2 | 41.44 | 0.2974 |
| 3 | 43.36 | 0.2688 |
| 4 | 48.18 | 0.3571 |
| 5 | 50.46 | 0.3498 |
| 6 | 66.00 | 0.2623 |
| 7 | 70.56 | 0.2860 |
| 8 | 74.14 | 0.3508 |
| 9 | 89.92 | 0.5161 |

1) **Cu layer**

Fig. 14 shows the analysis results.

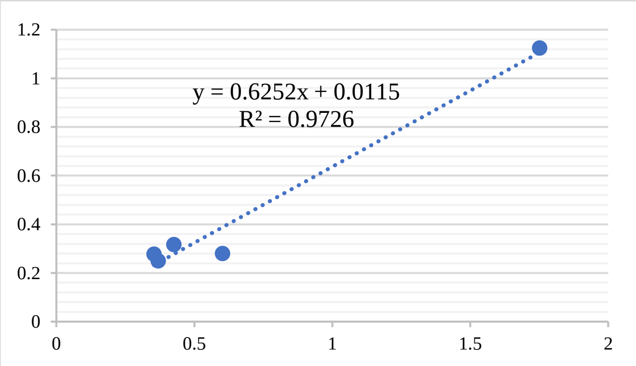


**Fig. 14.** Cu deposition voltage of 8 volts

Fig. 14 shows a graph of the linear equation resulting from the Cu layer at a deposition voltage of 8 volts. With the value obtained:

$y = 0.6252\,x + 0.0115$

And for the lattice strain value of the 8-volt Cu layer, it is 0.6252/4=0.1563 nm, so to determine the grain size, it can be calculated using the formulae:

$$b = \frac{K\lambda}{D}$$

$$0,0115 = \frac{0,9\times(0,15406\times10^{-9})}{D}$$

$$0,0115D = 0,9\times(0,15406\times10^{-9})$$

$$D = \frac{0,9\times(0,15406\times10^{-9})}{0,0115} = 1,17751\times10^{-8}\,nm$$

2) **Ni layer**

Fig. 15 shows the analysis results

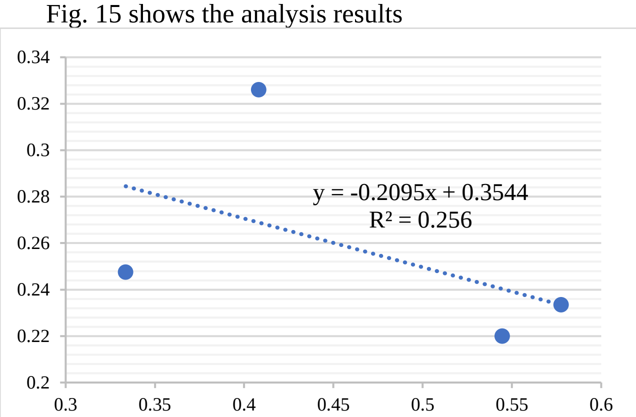


**Fig. 15.** Ni deposition voltage of 8 volts.

Fig. 15 shows a graph of the linear equation resulting from the Ni layer at a deposition voltage of 8 volts. With the value obtained:

$y = -0.2095\,x + 0.3544$

And for the lattice strain value of this 8-volt layer, it is (-0.2095)/4=-0.052375 nm, so to determine the grain size, it can be calculated using the:

$$b = \frac{K\lambda}{D}$$

$$0,3544 = \frac{0,9\times(0,15406\times10^{-9})}{D}$$

$$0,3544D = 0,9\times(0,15406\times10^{-9})$$

$$D = \frac{0,9\times(0,15406\times10^{-9})}{0,3544} = 3,82094\times10^{-10}\,nm.$$

After analyzing the data on samples with varying deposition voltage, the grain size of Cu can be determined as a whole.

$$\overline{D} = \frac{\sum_{i=1}^{N} D_i}{N} = \frac{2,50\times10^{-8}}{5} = 5,00\times10^{-9}\,nm$$

And for Ni is:

$$\overline{D} = \frac{\sum_{i=1}^{N} D_i}{N} = \frac{2,06\times10^{-9}}{5} = 4,13\times10^{-10}\,nm$$

See Table VI for all calculation result.

TABLE VI

DEPOSITION VOLTAGE VS. GRAIN SIZE

| Deposition Voltage (V) | Grain Size Nickel (Ni) |
|---|---|
| 6.0 | 2.03×10^(-10) |
| 6.5 | 5.05×10^(-10) |
| 7.0 | 1.57×10^(-10) |
| 7.5 | 8.18×10^(-10) |
| 8.0 | 3.82×10^(-10) |

Next, Table 20 presents the grain size values of Ni formed on the surface of the Cu layer due to the difference in applied voltage. The following will explain a graph regarding the relationship between deposition voltage and Ni grain size formed.

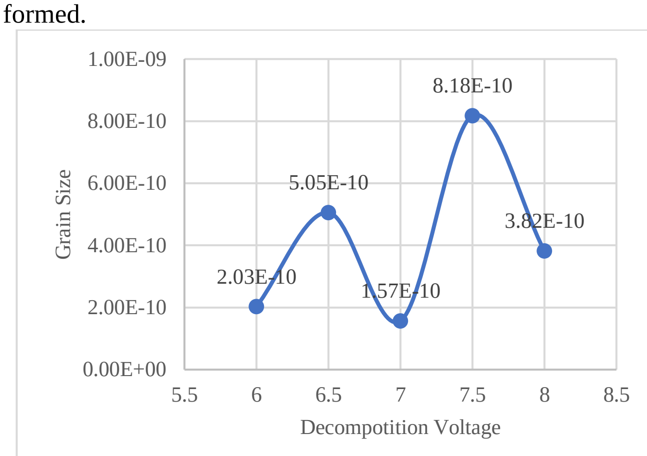


**Fig. 16.** Correlation between deposition voltage and grain size

From the above curve, it can be seen that there is an effect of side deposition voltage on the grain size of Nickel, where the resulting grain size fluctuates and is uncertain. This curve has the smallest grain size, namely when the applied voltage is 7 volts with a resulting grain size value of 1.57×10^(-10) nm. Optimum voltage also produces the largest grain size when Ni is given a voltage of 7.5 volts with a resulting grain size value of 8.18×10^(-10) nm.

## V. Conclusion

Based on the research results, the particle size of Cu and Ni layers with different applied voltages have different values due to various factors. The final value for the particle size of Copper (Cu) after analysis and finding the value of each particle size at varying voltages is 5.00 × 10^(-9) nm. Meanwhile, the overall particle size of Nickel (Ni) is 4.13 × 10^(-10) nm, and at an optimum voltage of 7.5 volts, Ni has a value of 8.18 × 10^(-10) nm. The applied voltage affects the particle size produced, and there is an optimum voltage at a deposition voltage of 7.5 volts.

## References


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