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Effect of Process Temperature on Nickel Powder Production using Electrodeposition Method in $NiSO_4$-$NiCl_2$-H_3BO_3 Solution

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Abstract

This paper aims to know the effect of process temperature on nickel powder production using the electrodeposition method in 500 ml of $NiSO_4$-$NiCl_2$-H_3BO_3 solution. The temperatures that were varied were 27°C, 40°C, and 50°C. The processing time was 15 minutes, and the current density was 2.2 A. The difference in the effect of temperature will be explained by the weight of the metal powder produced in the electrodeposition process. The powder results were then analyzed using an optical microscope, and the experiment that produced the highest powder weight would be further analyzed using a Scanning Electron Microscope (SEM) to characterize the morphology and grain size of nickel powder. The chemical composition was analyzed using Energy Dispersive X-Ray (EDX). The results show that the weight of nickel powder after electrodeposition is 0.58 g, 0.49 g, and 0.46 g for 27°C, 40°C, and 50°C, respectively. The most weights of nickel powder is produced from 27°C. The powder that is produced at 27°C has a smaller size and is uniform. The morphology of nickel powder is irregular with an average powder size range is 1- 5 μm. The higher current efficiency occurs at 27°C with the current density of 2.4 A/dm^2

Keywords: Electrodeposition, Current, Nickel Powder, Temperature

INTRODUCTION

The current technological development related to the metallurgical process is the government's support to increase the role of mineral processing and metallurgical companies to increase the independence and resilience of the national mineral-based industry from upstream to downstream. Powder metallurgy technology is a metallurgical downstream industry where most of the processes are printing processes for commercial workpieces or related to the process of forming finished products. Molded products made of metal are usually produced from a casting process using high temperatures. However, before the product can be used by consumers, many more steps are needed which take a lot of time. One of the products of the powder metallurgy process is metal objects or alloys in machine parts that have a high level of accuracy.

Casting results sometimes still have poor quality properties. Along with the time to improve the quality and efficiency of the process of forming the workpiece, the process of making components using a powder metallurgy process is carried out. One method of making metal powders is through the electrodeposition of metals from the extraction and purification processes.

In this research, the advanced application of powder metallurgy by providing a variable heat treatment process (temperature difference) during the electrodeposition process would be studied. The nickel-metal with high purity was used as the anode and stainless steel as the cathode. The metal to be decomposed functions as an anode and then the metal is made into powder by using an electric current in a 500 ml electrolyte solution which has a mixed composition of $NiSO_4$-$NiCl_2$-H_3BO_3 with temperature variations of 27°C, 40°C, and 50°C. The deposited powder will migrate attached to the cathode.

The advantage of making metal powders from electrodeposition results can be used to make product components with small sizes. In addition, when converted into finished products, not much powder material is wasted. The parameters of the success of this powder metallurgical formation process are to pay attention to the weight of the highest nickel powder and then further analyzed by optical microscopy and SEM-EDX to determine the morphology, grain size, and current efficiency.

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LITERATURE REVIEW

Process Powder metallurgy technology was well known around the 18th century. But at that time the most widely produced metals were gold and silver. Around 1870, powder metallurgy progressed to other metallic materials. Production of nickel powder through electrodeposition was introduced in the 1950s (Capson, 1951; Kuroda, 1953). Nickel is one of the metals known for its resistance to corrosion and its ability to withstand high temperatures without reducing its mechanical strength (Hsu et al, 2007; Walther et al, 2008). Electrodeposition of nickel using sulfate solution and addition of glycerol additive (Viswanath et al, 2013) has been studied. Nickel powder products, among others, are used to improve strength and quality properties in the automotive, aircraft spare parts (Nobrega et al, 2008), nickel-metal alloys (Sreenu et al, 2020), and the oil and gas industry (Alekseeva et al, 2021). These products are mostly produced through the casting process and must go through many stages of advanced processes to become finished products. After knowing the function of nickel, the powder metallurgical process became famous for being used as a raw material for 3D printing, thermal spray coating, and superalloys (Ryndenkov et all, 2017; El Rakayby & Kim, 2017).

In the electrodeposition process, a series of electrolysis equipment consisting of an anode and a cathode is required. The use of cathodes made from stainless steel 304 is one of the most widely used alloys and is often used for the electrodeposition process. The efficiency of the current that occurs at the cathode can be determined by applying Faraday's law. Faraday's law shows that the amount of metal deposited at the cathode and the amount dissolved at the anode are directly proportional to the amount of electric current passed. Faraday's law also relates the amount of a particular metal deposited or dissolved to its atomic weight and the number of electrons involved in electrochemical reactions. Faraday's law is used to calculate theoretical weights which will then be used to calculate current efficiency (CE). W is the theoretical weight of the powder (g), E is the equivalent weight (g), I is the current in amperes, t is the time in seconds.

$$W = (E.I.t)/96500$$

The current efficiency (CE) = (weight of experimental results)/ (theoretical weight) x100%

The process in the electrodeposition experiment is almost the same as the electroplating process, the quantity that needs to be considered is the current density, namely the current per unit surface area of the workpiece (Pujiyulianto & Suyitno, 2019). The cathode region in contact with the electrolysis solution will produce a powder that can be calculated by weight. The weakness of the electrodeposition process lies in the powder product which sometimes still contains surfactant residues that can contaminate the powder (Inazawa et al, 2008).

The characteristics of the powder produced from the nickel powder manufacturing process using the electrodeposition method will be greatly influenced by the process variables used, including the temperature during the electrolysis process. This research tries to make nickel powder produced by the electrodeposition method using a solution of $NiSO_4$-$NiCl_2$-H_3BO_3 with temperature variations of 27 °C, 40 °C, and 50 °C. The difference in the effect of temperature will be explained through the results of the weight of the metal powder produced. The powder results were then analyzed with an optical microscope, SEM-EDX, and calculated the theoretical weight using Faraday's Law to determine the current efficiency.

RESEARCH METHODOLOGY

Electrode Material

The electrodes used in this electrodeposition process consist of an anode and a cathode. The anode used is a nickel-metal plate purchased online imported from Sumitomo Metal Mining, Japan with a purity level of Ni>99% (figure 1.a). The cathode used is a 304 stainless steel plate with a size of 50 mm x 90 mm x 0.1 mm. This stainless steel has a composition of 8.1%Ni, 18.5%Cr, 0.042%C, 1.19%Mn, 0.043%P, 0.003%S, 0.530%Si, and the rest is Fe. The area of the cathode in contact with the solution is 0.9 dm2 (figure 1.b).

Electrolyte Solution

The electrolysis solution used is a combination of Nickel Sulfate solution with a mixed composition of $NiSO_4$ 250.19 g/l, $NiCl_2$ 63.94 g/l, H_3BO_3 51.65 g/l. Electrolyte solutions contain electrically charged particles or ions. This electrolysis solution is green with a pH of 4.8 (figure 1.c)

Hot Plate Magnetic Stirrer

Hot Plate Magnetic Stirrer (Thermofisher, US) is used to create temperature conditions for the electrodeposition process according to predetermined variables. The temperature of the solution is set to reach a constant of 27°C, 40°C, and 50°C. Hot Plate Magnetic Stirrer is equipped with a magnetic rod as a stirrer (figure 1.d).

Rectifier

Rectifiers are used to provide electric current. The current used for the process is 2.2 Ampere. This PS- 40 AR-type rectifier has a maximum voltage of 15 V and a current of 50 A (Figure 1.e).

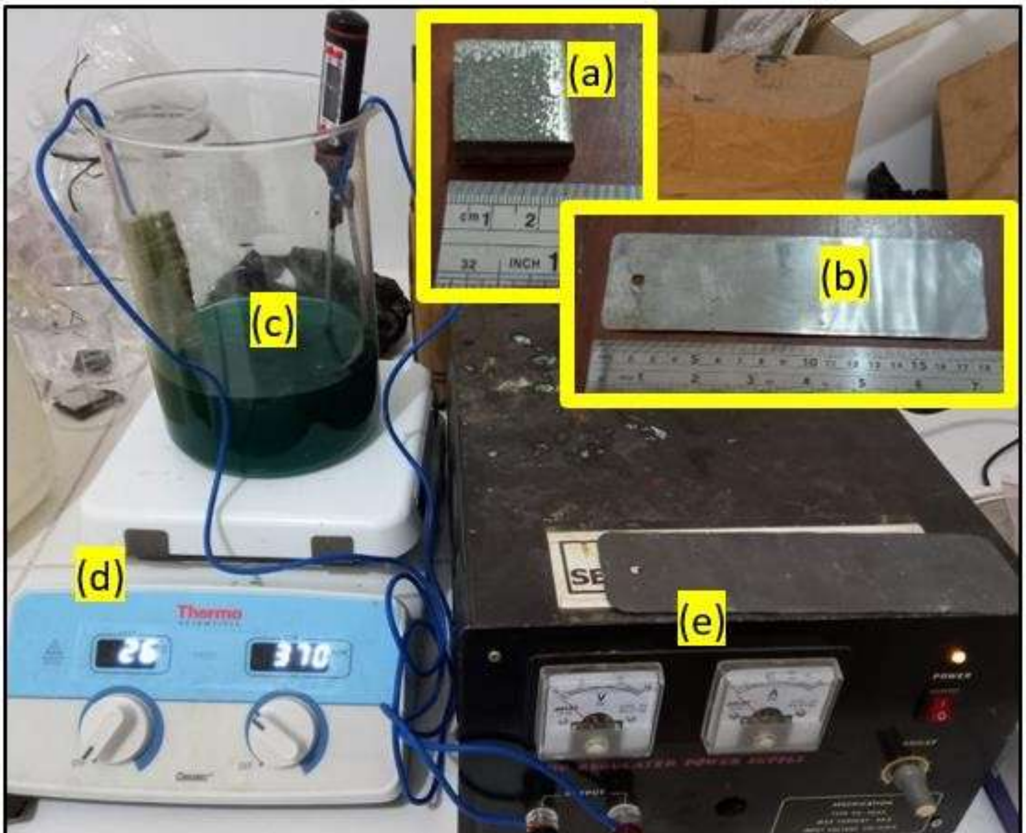


Figure 1. Experimental tools and materials. High purity nickel anode (a), Stainless steel cathode 304 (b), Electrolysis solution $NiSO_4$-$NiCl_2$-H_3BO_3 (c), Hot Plate Magnetic Stirrer (d), rectifier (e)

Electrodeposition process

The electrodeposition process was carried out at a solution volume of 500 ml in a 1 L pyrex glass beaker. The cathode and anode are immersed in the electrolyte and connected by copper wire to the rectifier. The experiment was carried out for 15 minutes. At the cathode will attach nickel powder that has migrated to stainless steel. Remove the cathode and dry it in the open air. Extraction of nickel powder attached to the cathode through scraping or striping to further weigh the weight of the nickel powder taken. Nickel powder was weighed using an analytical balance.

Study of morphology and grain size

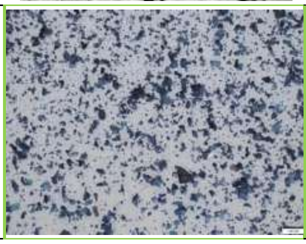
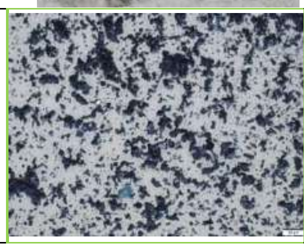
Nickel powder was characterized microscopically (Olympus U-MSSP4, Japan) and SEM-EDX (Hitachi SU-3500 and JSM-IT200, Japan). Photomicrographs were analyzed to determine powder morphology and grain size. EDX was used to analyze the composition of nickel powder formed.

FINDING AND DISCUSSION

Finding

After the electrodeposition process is complete, to get the actual weight of the powder, it must go through drying first so that the resulting powder is completely dry. After that, the grinding test was carried out with the help of ceramic mortar for 15 minutes. Nickel powder was successfully collected with an actual weight of 0.58 g, 0.49 g, and 0.46 g at each variable temperature of 27°C, 40°C, and 50°C. The macro photo of nickel powder shown in table 1 shows the color difference between the three processes. However, overall it shows that the nickel powder production process using the electrodeposition method with a variable temperature difference has been successfully carried out. The difference is indicated by the presence of electrolysis residue deposits that are strongly attached to the resulting powder attached to the cathode. The use of an optical microscope with a certain magnification produces nickel powder which has a different physical appearance. At room temperature (27°C), a powder with a smaller and uniform grain size was produced compared to the powder at 40°C and 50°C. The physical appearance of nickel powder at 40°C resulted in larger grain size and a dark green residue layer. Likewise, for the powder results in the 50°C process, larger and less uniform grain size powders were produced. In the powder, there is still a residue of electrolysis solution deposition.

Table 1. Experimental results based on the difference in temperature of the electrodeposition process.

Parameter	Variable Temperature		
	27 °C	40 °C	50 °C
Powder yield			
Macro results Structure With optical microscope			
Physical appearance	The grain size is smaller and uniform.	The grain size is larger and there is a residue layer.	The grain size is larger and there is a residue layer.
Powder Weight	0.59 g	0.49 g	0.46 g

Current Efficiency	96.33 %	81.38 %	76.40 %
Theoretical Weight	0.6 g		

Current efficiency is the ratio of actual weight to theoretical weight. If the actual weight is closer to the theoretical weight, it will produce a high percentage of current efficiency and vice versa. The higher current efficiency is 96.33% resulting from the electrodeposition process at a temperature of 27°C. In this condition, smaller and more uniform nickel powder can be produced. Based on table 1, the effect of temperature changes on the electrodeposition process produces different current efficiencies, and also shows that the higher the temperature used, the smaller the current efficiency. Calculation of the theoretical weight of 0.6 g is calculated using the formulation of Faraday's law. The results in Table 1 show that the electrodeposition process at room temperature (27°C) has a smaller and uniform powder size than the others.

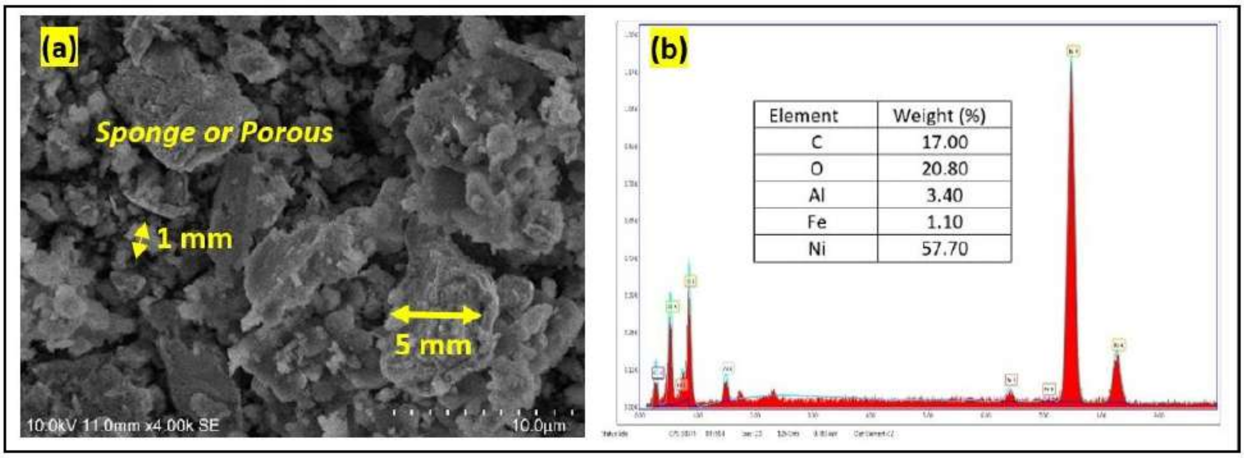


Figure 2. SEM-EDX photomicrograph image, nickel powder produced using electrodeposition at 27°C (a), chemicals contained in nickel powder at 27°C electrodeposition process (b)

The results of photomicrographs using SEM showed a very clear form of nickel powder. The morphology of nickel powder is irregular to form a sponge with an average powder size of 1-5 micrometers and is porous (figure 2.a). The chemical composition of nickel powder is 17%C, 20%O, 3.4%Al, 1.1%Fe, 57.7%Ni (figure 2.b).

DISCUSSION

The working principle of electrodeposition is the process of forming metal powder using an electric current through an electrolyte solution. At the anode when given a current, nickel will release electrons to form NI^{2+}, which then dissolves in the electrolysis solution, the electrons released will flow towards the cathode, when they arrive at the cathode, the electrons around the cathode will be recaptured by Ni^{2+}, resulting in a reduction reaction resulting in Ni. The reduced Ni will move towards Fe and will coat the Fe, in this case, stainless steel. Giving the right current & voltage will make the anode decompose/dissolve in the electrolysis solution. Through this electrolyte solution, the dissolved metal will be deposited on the cathode. The results of this nickel powder will later become the success of the next workpiece process. The manufacture of a product will be greatly influenced by the shape, size, and other parameters that exist in the powder used.

The smaller the particle size, the larger the surface area and better mechanical properties. In the experiment, smaller and uniform particle sizes were obtained in a process that took place at room temperature (27°C). At 27°C, the cleanliness of the powder against the residual deposition of the electrolyte solution is cleaner than others. In the electrodeposition experiment at 40°C and 50°C, there was a residue on the cathode which was indicated by a different colored precipitate. These deposits include nickel powder deposits, green residue deposits, and black residue deposits. This residue deposit is a salt coating mechanism on the electrolyte solution which will contaminate the final powder product. The chemical composition of the powder contains iron and carbon impurities. The Fe content can be in the form of inclusions from the stainless-steel cathode during the nickel powder extraction process, while the presence of carbon is possible due to the high process temperature, so there may be parts of the electrolysis solution that react to form carbon elements.

Current density affects the current efficiency, weight, morphology, and grain size of nickel powder. The shape of the powder particles will affect the packaging of metal powders and compressibility during the workpiece manufacturing process. The current density in the three experiments was the same, but due to the process variable, the temperature increases also affected the flow efficiency, weight, morphology, and grain size of nickel powder. The effect of temperature on the actual weight of the powder and the flow efficiency is shown in Figure 3, it is shown that at a temperature of 27°C it produces the most powder (0.58 g), and the highest flow efficiency level is also (96.33%).

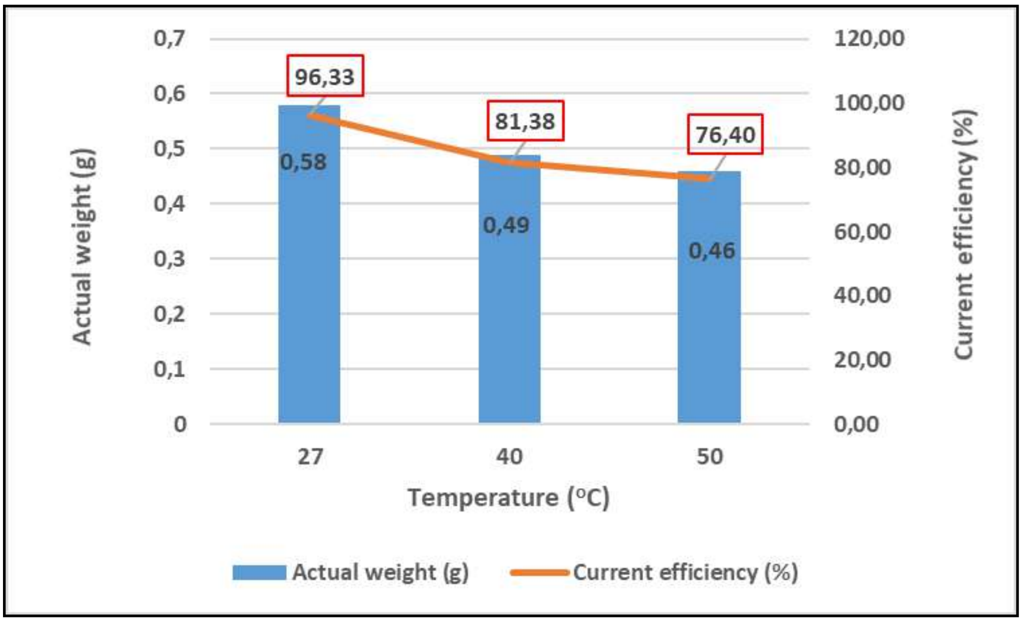


Figure 3. Effect of temperature on the actual weight of the powder and current efficiency

CONCLUSION AND FURTHER RESEARCH

Conclusion

The effect of process temperature on nickel powder production using electrodeposition method in 500 ml of $NiSO_4$-$NiCl_2$-H_3BO_3 solution have been conducted, and the conclusions are as follows: The results show that the weight of nickel powder after electrodeposition are 0.58 g, 0.49 g, and 0.46 g for 27°C, 40°C, and 50°C, respectively. The most weights of nickel powder is produced from 27°C. The powder that is produced at 27°C has a smaller size and is uniform. The morphology of nickel powder is irregular with an average powder size range is 1-5 µm. The higher current efficiency occurs at 27°C with a current density of 2.4 A/dm^2.

Further Research

The suggestions for further research are:

- It is necessary to do further research with different variables, including the use of higher currents, the type, and composition of the electrolysis solution, pH, agitation speed, operating time, the concentration of electrolysis solution, and the addition of additives.
- Immediately follow up on further research to provide maximum benefits for the development of the domestic manufacturing industry.

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REFERENCES

Alekseeva, E., Shishkova, M., Strekalovskaya, D., Gerashchenkov, D., & Glukhov, P., (2021), Ni-Based Coatings for Oil and Gas Industry Fabricated By Cold Gas Spraying. Int. Mater. Proc. 3 (1)

Copson H.R., A. Wesley, and T.H. Wickenden, (1951), Electrolytic Production of Nickel Powder, Canada Patent No.,475/886

El Rakayby H., Kim K. (2017), Deformation and Densification Behaviours of Nickel-Based Superalloy During Hot Isostatic Pressing, Powder Metall., 60 (4) 293-300

Hsu, C. H., Chen, M. L., & Hu, C. J., (2007), Microstructure and Mechanical Properties of 4% Cobalt and Nickel Alloyed Ductile Irons, Mater. Sci. And Eng., 444 (1-2) 339-346.

Kuroda M, (1953), Electrolytic Preparation of Nickel Powder, Japan Patent No. 5166/53

Nobrega, B. N., Ristow, Jr, W., & Machado, R., (2008), MIM Processing and Plasma Sintering of Nickel Base Superalloys for Aerospace and Automotive Applications. Powder. Metall., 51(2) 107-110.

Pujiyulianto Eko & Suyitno, (2019), The Effect of Electropolishing Time Variation at Room Temperature and Low Voltage on The Surface Quality of Cardiovascular Stent, Adv. Mater. Reas., (2019) (1154),91-101.

Ryndenkov D. V, Et All. (2017), Rheological Properties Of EP962NP Nickel Powdered Superalloy Under Deformation In The Two-Phase Region With Industrial Stamping Rates And Structural Changes Corresponding To Deformation, Russian J. Of Non-Ferrous Metals, 58 (2) 136-141.

Sreenu, B., Sarkar, R., Kumar, S. S., Chatterjee, S., & Rao, G. A., (2020), Microstructure and Mechanical Behaviour of An Advanced Powder Metallurgy Nickel Base Superalloy Processed Through Hot Isostatic Pressing Route for Aerospace Applications, Mater. Scie. And Engg.: A, 797

Viswanath S.G., Manish M. Jachak, (2013), Electrodeposition of Nickel Powder from Sulphate Solution In Presence Of Glycerol And Sulphuric Acid, Assoc. Metall. Engineers. Serbia. 19 (3) 233-248

Viswanath S.G., Sajimol George, (2013), Electrowinning of Nickel Powder from Glycerol and Ammonical Medium- Study of Morphology and Nano-Nature of The Powder, Assoc. Metall. Engineers. Serbia, 18 (2) 129-143.

Walther, G., Klöden, B., Büttner, T., Weissgärber, T., Kieback, B., Böhm, A., & Timberg, L, (2008), A New Class of High Temperature and Corrosion Resistant Nickel-Based Open-Cell Foams. Adv. Eng.G Mater., 10 (9) 803-8.