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Nickel and Nickel Composite Electroplating from Nickel Sulfamate Bath with SiC Nanoparticles

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Abstract

Nickel is a metal that has been included in new research and development in engineering fields due to its properties and widespread use in the industry. Thanks to its great resistance to corrosion and abrasion, it can be used in several industries. The best way to increase the mechanical properties and surface quality of the produced material with an external effect is coating. Composite coatings are made to increase the properties of the coating made in line with the needs of the developing technology and industry. There are matrix and reinforced materials that are needed for composite coating. Nickel provides suitable properties as a matrix material due to its properties and silicon carbide is used since it is one of the hardest reinforced materials. In this study, nickel composite coatings were produced from nickel sulfamate electroplating bath containing different silicon carbide nanoparticle amounts. Samples were examined to increase their hardness with the effect of silicon carbide.

Riassunto

Il nichel è un metallo che è stato incluso in una nuova ricerca e sviluppo nel campo dell'ingegneria grazie alle sue proprietà e al suo uso diffuso nell'industria. Grazie alla sua grande resistenza alla corrosione e all'abrasione, viene utilizzato in diversi settori. Il modo migliore per aumentare le proprietà meccaniche e la qualità superficiale del materiale prodotto con un effetto esterno è il rivestimento. I rivestimenti compositi sono realizzati per aumentare le proprietà del rivestimento realizzato in linea con le esigenze della tecnologia e dell'industria in via di sviluppo. Ci sono matrici e materiali rinforzati sono necessari per il rivestimento composito. Il nichel fornisce le proprietà adatte come materiale di matrice grazie alle sue proprietà e viene utilizzato il carburo di silicio poiché è uno dei materiali rinforzati più duri. In questo studio, i rivestimenti compositi di nichel sono stati prodotti da un bagno di galvanica al solfammato di nichel contenente diverse quantità di nanoparticelle di carburo di silicio. I campioni sono stati esaminati per aumentare la loro durezza con un effetto del carburo di silicio.

1. INTRODUCTION

Material properties are determined by internal structure, chemical composition, and productive way and besides, they are affected by exposure environment conditions, working conditions, and duration time.

Increasing needs to enhance and a demand-supply gap in industry prompt scientists and engineers to find new and economical ways to produce a material which has better mechanical property, long service life, and good appearance. Developments of technology in engineering applications give a possibility to produce new materials or enhance the properties.

Corrosion and wear resistance of materials are important properties for their durability and lifetime so research about improving corrosion and wear resistance of materials gain priority in engineering areas. In order to cope with these problems and develop research, coating methods play a major role in the protection and durability of materials.

The coating is a protection method from corrosion and material gain resistive against wear so material durability increases underexposure conditions. In some cases, the coating helps to improve the appearance of the material and enhance surface condition.

Coating material should be chosen properly according to usage area and aim of material to avoid the high cost and unnecessary energy consumption and extra raw material consumption during the process.

1.1. History

The first coating method which people used is painting. At those times, unlike today, a coating made just only decorative purposes as painting a figure on stones using pigments in the stone age. In the history of humanity, the first protective coating belongs to Egyptians. They used pitch and balsam for basic waterproofing to seal boats (Raymond R., 1981).

In 1805, Luigi V. Brugnatelli took the first step of electroplating by connecting metal and gold with a voltaic battery inside the solution which was prepared with dissolved gold. After his experiment, small materials started to be plating. After the invention of silver and gold plating, 19th-century plating was used for good, smooth, and shiny surfaces (Engineering and Technology History, 2015).

Electroplating became important and started using smart, functional, and protective coatings. In 1920, George J. Sargent published his work about chromium electroplating (Dubpernell, 1960). Chromium, which has been used for a long time in coatings for increase material strength, was banned in Europe in 2003 because of its damages that caused to the environment and human health (Dubpemell, 2012).

After research to find an alternative coating as strong as chromium, Nickel and its salt solutions became an indispensable and important metal thanks to its high wear resistance, corrosion resistance, and workable due to hard and ductile structure at plating and decoration fields (Torabinejad, Aliofkhazraei, Assareh, Allahyarzadeh, & Sabour Rouhaghd, 2016). After comparing the pure metals or alloys with the electro-co-deposited metals or alloys, it is seen that the corrosion and wear resistance are improved in electro co-deposited metals or alloys (L. Wang, 2005). Having nano-size articles in the 1990s results from the increment in properties of coatings and this makes the interest to Ni-based nano-composite coatings higher (Pavlatou, Stroumbouli, Gyftou, &., 2006).

Nickel and its different salt solutions are using to make the coating as a protective layer on the surface by electrodeposition, electroless deposition, thermal spraying, weld surfacing, pulsed-current plating, and chemical vapor techniques. In general, electrochemical techniques for coating are preferred because of low cost, easy control, and versatility. As it knows, electrochemical techniques can be easily modified for the desired coating while adhering to its traditional techniques and electrodeposition setup enable easy to control parameters and different elements can be added to electrodeposition bath to gain different properties for coating (Birlik & Ak Azem, 2018).

There are several types of nickel salt solutions which are Watts Nickel solution, Nickel sulfamate solution, All-chloride solution, Sulfate-Chloride solution, All-Sulfate solution, and Hard Nickel solution. Solutions are choosing according to purposes. Nickel sulfamate solutions are widely preferred in terms of industry advantages and usability.

Table 1 - Nickel electroplating Solutions *(Kopeliovich, 2013)*

Solutions	**Bath Compositions**	**Operating Conditions**		
		Temperature	Current Density	pH
Watts Solution	Nickel Sulfate ($NiSO_4H_2O$): 240-300 g/L	40 - 65 °C	2 - 10 A/dm²	3.0 - 4.5
	Nickel Chloride ($NiCl_2.6H_2O$): 30-90 g/L			
	Boric Acid (H_3BO_3): 30-45 g/L			
Nickel Sulfamate Solution	Nickel Sulfamate ($NiSO_4.6H_2O$): 300-450 g/L	40 - 60 °C	2 - 25 A/dm²	3.5 - 4.5
	Nickel Chloride ($NiCl_2.6H_2O$): 0-30 g/L			
	Boric Acid (H_3BO_3): 30-45 g/L			
All-Chloride Solution	Nickel Chloride ($NiCl_2.6H_2O$): 225-300 g/L	43 - 65°C	2.5 - 10 A/dm²	1.0 - 3.0
	Boric Acid (H_3BO_3): 30-35 g/L			
Sulfate-Chloride Solution	Nickel Sulfate ($NiSO_4H_2O$): 150-225 g/L	43 - 52°C	2.5 - 15 A/dm²	1.5 - 2.5
	Nickel Chloride ($NiCl_2.6H_2O$): 150-225 g/L			
	Boric Acid (H_3BO_3): 30-45 g/L			
Fluoborate Solution	Nickel Fluoborate ($Ni(BF_4)_2$): 225-300 g/L	38 - 70°C	3 - 25 A/dm²	2.5 - 4.0
	Nickel Chloride ($NiCl_2.6H_2O$): 0-15 g/L			
	Boric Acid (H_3BO_3) : 15-30 g/L			
All-Sulfate Solution	Nickel Sulfate ($NiSO_4H_2O$) : 225-400 g/L	38 - 70 °C	1 - 10 A/dm²	1.0 - 4.0
	Boric Acid (H_3BO_3) : 30-45 g/L			
Hard Nickel Solution	Nickel Sulfate ($NiSO_4H_2O$) : 225-400 g/L	43 - 60°C	2.5 - 5 A/dm²	5.6 - 5.9
	Ammonium Chloride (NH_4Cl_3) : 25 g/L			
	Boric Acid (H_3BO_3) : 30 g/L			

First sulfamate plating solutions including nickel published in 1930 by R. Piontelli and L. Cambri. The first U.S. patent for sulfamate plating covering nickel belongs to M. E. Cupery in 1943. In 1950, Richard described nickel sulfamate coating, which has been used in the electrical industry since 1949 (Baudrand, 1996).

To make a strong and resistive nickel alloy coating as chromium, a second hard phase is needed to improve the properties. Carbides nanoparticles are the most used one among the ceramic, oxides, or metal particles.

1.2. Purpose

The main aim of this project to observe the effect of silicon carbide (SiC) nanoparticles on hardness and nucleation at nickel sulfamate electrodeposition bath. Different compositions which contain different amount of SiC nanoparticle were studied to find a suitable composition for getting better hardness results. Hardness results of deposition by nickel sulfamate solution bath without contain SiC nanoparticle were compared with hardness results of depositions by four different nickel sulfamate solutions which contain different amount of SiC nanoparticle bath. Each experiment runs three times to proof the values and after results, two more solutions which contain different amount SiC nanoparticle were prepared to compare hardness results. In this way, a suitable and economical nickel sulfamate solution including SiC nanoparticle was determined for a solution that has an acceptable hardness value containing less amount of SiC nanoparticle. Besides, some samples were examined with AFM to observe the roughness of surfaces on cupper wafer deposition.

1.3. Delimitations

The thesis is only focused on the effect of one type of nanoparticle silicon carbide which is a specific particle size 100nm with electroplating. The composition of nickel sulfamate solution is kept constant with varying different amounts of nanoparticle amount to compare hardness results. In addition to this, for all samples, experimental parameters are defined inside standard values and kept constant.

2. THEORETICAL BACKGROUND

Coating or covering the surface partially and totally can be done by 2 basic deposition methods which are electroless deposition and electrodeposition from a deposition point of view.

Electrodeposition is that the surface of metallic or non-metallic materials is cover with a metallic layer on top of material by electrochemical methods. Today, electrodeposition is a necessary process which we need for both daily usage material that is used watch and glasses and industrial areas like car, plain parts, or electronic device. Electroplating brings many advantages to engineering applications. Mainly, it provides corrosion resistance and wears resistance and easy procedure, production, and low cost.

2.1. Electroless deposition

Electroless deposition is a coating method that runs by chemical reactions instead of current flow. In this technique, the coating layer is formed by a chemical reaction as known autocatalytic reaction. Unlike electrodeposition which uses current to move the particles or ions in order to form a coating layer, inside bath chemical reducing agents which are inside the bath solution reduce the metal ion to deposit on the top of the surface. The surface should be a catalytic for successful surface finish otherwise, catalytic reduction reaction cannot run which is needed.

Electroless deposition bath consists of a metal source, reducing agent, surface-active agents, buffer, chelating agents, accelerators, and stabilizers. Metal sulfates use as a general metal source. For deposition, process pH is an important parameter, and it plays a major role in the reaction. Buffers are used to control the pH value of the reaction and they keep constant to prevent harmful side reactions due to hydrogen ions which are produced during the process. Chelating agents help to keep the metal inside the solution to complete the reduction reaction. Stabilizers keep under control the autocatalytic reaction rate while accelerators increase the speed of precipitation on the surface.

$$METAL\ ION(M^{+}) + REDUCING\ AGENT \rightarrow METAL(M) + OXIDISED\ PRODUCT$$

Reducing agents inside the solution bath oxidize and release electrons. Metal ions take these electrons, and they reduce to become metal which is deposited on the surface.

The major reason why it is preferred that since electrical flow is not used, insulant materials as plastics can be covered by this technique as well as metals. For complex shape materials and surfaces which are hard to reach as cavities can be covered with uniform and low porosity thin layer. Material gains unique chemical and mechanical and magnetic properties with a coating layer, and these contributions increase the durability of corrosion and wear resistance.

There are three techniques for an electroless deposition that are immersion plating, homogenous chemical reduction, autocatalytic deposition (Interplex Holdings Pte. Ltd., 2018).

2.1.1. Immersion Plating

By immersion plating, the more noble metal is deposited on a less noble metal surface. Immersion plating has narrow application areas since the deposited layer is thin and non-adherent coatings. The common application is that steel is immersed in the copper solution, and copper ions that are inside the solution are deposited on the steel surface (Interplex Holdings Pte. Ltd., 2018).

2.1.2. Homogenous Chemical Reduction

There is a chemical reagent that generates the electron to reduce the metal ions, and in this way, metal ions are deposited on the substrate surface. Bonding of the coating to the surface of the material is one of the disadvantages though the coating layer is thick. Another disadvantage of the homogenous chemical reduction method is that metal ion solution and chemical reagent react as soon as so, they should keep away from each other (Interplex Holdings Pte. Ltd., 2018).

2.1.3. Autocatalytic Deposition

In the autocatalytic deposition, chemical reducing agents are used to provide the electrons for plating. During the process, ions that are in the solution are settled to deposit onto naturally catalytic surfaces. The reaction is catalytic; therefore, the reaction is self-developed. As a result, the coating can have a significant thickness and be highly adherent (Interplex Holdings Pte. Ltd., 2018).

2.2. Electrodeposition

The electrodeposition process contains two electrodes into an electrolyte bath by applying an electrical current. This process occurs in an electrolyte bath which contains ions or electrically charged particle. Electrodes called anode that is positively charged, and cathode which is negatively charged immersed into the electrolyte. In general, an anode made from coating material and substrate which is coated takes place as cathode.

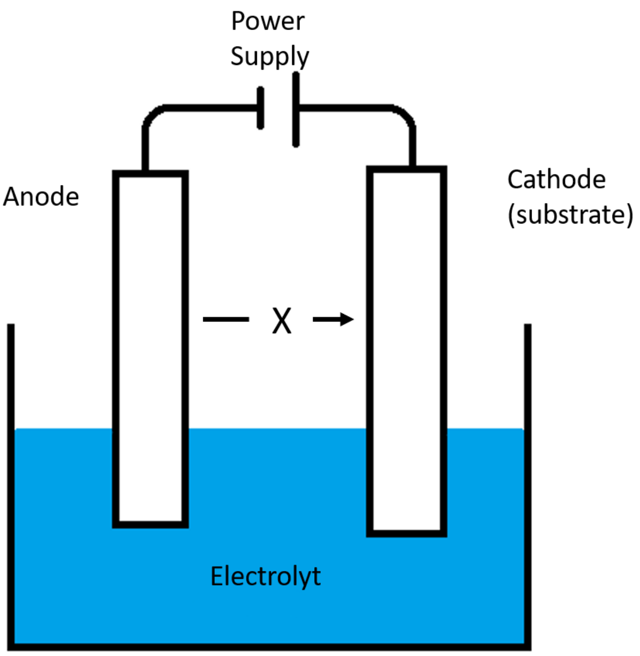

Figure 1 - Basic Demonstration of Electroplating

The substrate surface is covered by a metallic or alloyed layer using electrodes under electric current. When the voltage is applied between anode and cathode, ions start to move towards the oppositely charged side concerning their charge so, negatively charged ions move to the anode, and positively charged ions move to the cathode. In this way, the electrical circuit is complete with a current flow which is created by electron transfer (Nickel Institute, 2014). Anode and cathode are immersed in the electrolyte. Plating starts with the help of electrolytes when applying external current due to potential differences between electrodes. During the process, anodic material starts to dissolve with the help of ions in the electrolyte and substrate covers. In some cases, the anode is not soluble although it must be conductive, so the substrate covers by metallic ions in the electrolyte.

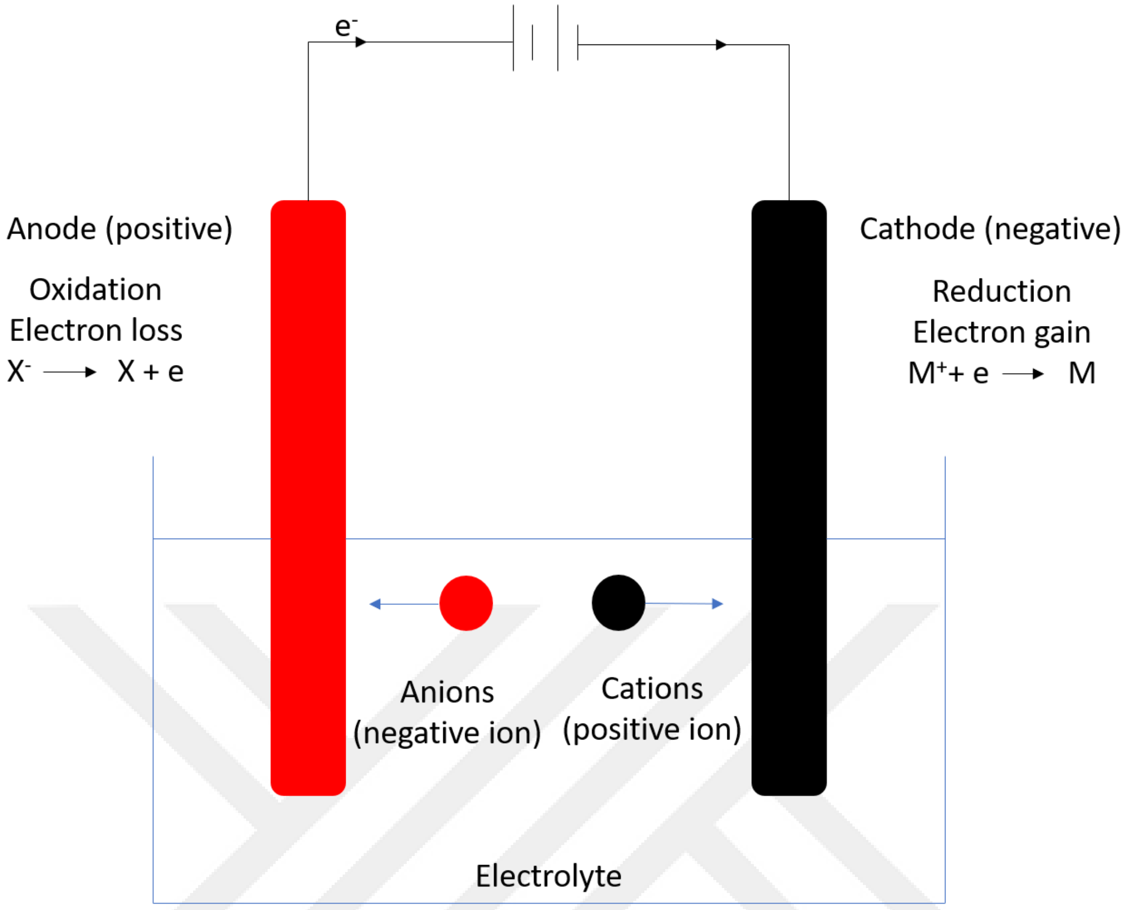

Figure 2 - Electron and Ion Movements

Insoluble anodes use to reduce cost in the electroplating industry. The main reason for the use of insoluble anodes is cost. Unlike soluble anode, the insoluble anode does not dissolve during the process. The ions inside the electrolyte deposit onto the substrate instead of the soluble anode. After complete deposition, the deposited layer has the characteristics of electrolyte ions. Therefore, electrolyte should prepare according to intended properties (Menard, 2006).

During the plating process, anode dissolution can cause undesirable side products, so it brings a waste of anode and an increase in cost. Insoluble anodes provide many advantages. First, insoluble anodes do not consume, and their service life is so long therefore, they can use for a long-time electroplating bath. Also, their geometries stay constant, and this helps to optimize metal distribution. Moreover, insoluble anodes eliminate potential problems comparing soluble anodes (Menard, 2006).

Electroplating is a cost-effective method to improve the properties of the material. Material is protected by two mechanisms that are sacrificial protection and mechanical protection. In sacrificial protection, the metals which are zinc or cadmium cover and protect the material because these metals are more reactive than the material itself. In mechanical protection, metals such as copper, nickel, chromium, tin, and most other metals provide mechanical protection, while the coating remains intact. If there are any defects in the plating layer, the substrate will corrode before the plating does (Interplex Holdings Pte. Ltd., 2018).

Electroplating includes four main stages that are cleaning, undercoat deposit, final deposit, and inspection. Each of these stages is common to all types of deposition. Cleaning is vitally important; without adequate cleaning and pre-treatment of the substrate, deposits will have adhesion problems. Undercoats and final deposits are usually specified to meet the respective specifications that are necessary to provide the functionality to the finished component and is very rarely discussed with the finisher at the design stage (Interplex Holdings Pte. Ltd., 2018).

2.2.1. Influence of Operation Parameters of Electrodeposition

Electrodeposition process depends on materials and solution which are in use set up and also environmental conditions (Kumar, Pande, & Verma, 2015).

2.2.1.1. Current Density and Distribution

Current density and distribution have a big effect on the uniform coating. The current density of the cathode is different for all points. During the deposition process, the current tends to flow from the nearest point to the farthest point of the opposite electrode to collect the edges of the substrate. Therefore, cathodic current density collects on edges of the substrate, but it decreases from edges to inside, vias and cavities. The current density of the cathode must vary inside proper interval values concerning both composition and temperature. The lower current causes poor coating besides excessive current increases deposition rate but it causes other difficulties to control parameters. When current density exceeds limiting interval values, hydrogen ions discharge, and it increases the pH of the cathode. Rising pH causes metal hydroxide ions, and the coating contains these ions. Anode current density is also an essential parameter, and it should be under control properly with depends on the total anode area and anode material. Eventually, current densities depend on the composition of the electrolyte, type of coating, operational conditions (Kumar, Pande, & Verma, 2015).

2.2.1.2. pH

Salt composition determines the pH of the electrolyte. pH value varies different values depends on coating. In some cases, salt cannot reach the desired pH value so to make efficient plating, acidic or basic salts are added to the bath and these salts choose depends on bath composition. During plating, oxygen evaluation takes place in the anodic region when hydrogen evaluation happens at the cathodic side. Between these two reactions, the pH of the bath changes with time, and these changes cause defects for example, because of hydrogen evaluation, co-deposited plate metal, and defective coating occur. Buffers are used to prevent the changing of pH (Kumar, Pande, & Verma, 2015).

2.2.1.3. Temperature

The temperature has a different effect on a deposition. Increasing the temperature of the bath enhances solubility so, the conductivity of the solution increases. Besides, high bath temperature causes the crystal size of the deposit, and it decreases the viscosity of the solution thereby, after the first layer of plating, other layers of plating deposit easily. Temperature also affects the mechanical properties of the coating. Adsorption of hydrogen reduces at a high-temperature bath; hence stress decreases, and cracks do not occur. From 45 °C to 55 °C, the grain size of the deposit decrease but increasing from 55 °C has the opposite effect (Kumar, Pande, & Verma, 2015).

2.2.1.4. Bath Concentration

Bath concentration changes the property of plating and affects the performance of the plating. Deposition rate enhances due to increasing concentration of metal ions at a solution (Kumar, Pande, & Verma, 2015).

2.2.1.5. Plating Time

Coating thickness depends plating time and applied current. The relation between plating time, current and thickness of deposition:

$$Q = I \times t$$

where, Q is charge flow, I is current flow and t is deposition time (Kumar, Pande, & Verma, 2015).

2.2.1.6. Agitation

Agitation affects the structure of the deposit indirectly. Agitation velocity should vary in interval and proportional to current density. First, it helps the mixing and dissolution of salts to make a homogeneous solution. Also, it reduces gas bubbles during plating so prevent surface defects as pits. It permits a higher current density operation and furthermore, during deposition, metal ions consume, and they replace rapidly with the help of agitation. However, agitation can cause coarse grain structure due to sludge or impurities inside the electrolyte (Kumar, Pande, & Verma, 2015).

2.2.1.7. Nature of Metal

According to electrochemical polarization, metals can divide into three groups. First groups like Ag, Pb, Cd, etc., deposited at overpotential and rough surface get obtained with grain size bigger than 10-3cm. Another group like Ni, Co, Fe, etc., has large electrochemical polarization and is deposited densely with 10-5cm of grain size. The last group contains intermediate metals like Cu, Zn, Bi, etc., and their properties vary between these two groups. Interaction of metal and coating layer material always different so, kinetic of deposition and grain size of the coating layer are different for every plating solution (Kumar, Pande, & Verma, 2015).

2.2.2. Purposes of Electrodeposition

Electroplating applies for different purposes. We can divide four main categories that are overlapping with each other.

2.2.2.1. Appearance

From the ancient time, a coating made because of appearance for decorative purpose. This kind of electroplating is made also for protective purposes but if the material will use inside not exposed to outdoor conditions such as a corrosive environment, the material coats the thin layer. Especially, gold deposited at a very thin layer to small pieces to have a shiny and bright surface (Kumar, Pande, & Verma, 2015).

2.2.2.2. Protective

The coating is carried to prevent wear and corrosion that are the main reasons for damage. A protective coating can apply in several ways by a range of different processes, as electrodeposition, electroless plating, thermal spray, weld surfacing, pulsed-current plating, and chemical vapor techniques. All these techniques have their benefit and drawback. In this study, electrodeposition is only considered a method (Kumar, Pande, & Verma, 2015).

2.2.2.3. Special Surface Properties

Some material needs some special treatments for their usage areas (Kumar, Pande, & Verma, 2015).

2.2.2.4. Engineering or Mechanical Properties

Some processes are carried out to increase the mechanical properties and the endurance of material to have a long service life. These processes can be an internal process such as changing material structure or composition or it can be external processes such as coating (Kumar, Pande, & Verma, 2015).

2.3. Nickel Electroplating

Nickel is an essential metal that is used in many industrial fields in the world thanks to its contribution to material due to its nature. At nickel electroplating, a thin layer of nickel has covered the substrate by electroplating.

Decorative, electroforming, and engineering applications are the main aims to use the nickel electrodeposition (Di Bari).

Nickel plating is applied to provide corrosion, improve wear resistance, strengthen obsolete materials, salvage small size materials, and decorative purposes.

Decorative applications take place for about 80% of the nickel consumed in plating, and the rest of the 20% is for engineering and electroforming. The 12 percent of nickel consumed annually in the world is used for electrodeposition. Nickel consumption for electrodeposition is 11% to 12% of world consumption that is approximately 180 million pounds (Di Bari).

Table 2 - Nickel Salts and their nickel contents *(Di Bari)*

Nickel Salts	**Formula**	**Content of Nickel (%)**
Nickel Chloride	$NiCl_2{\cdot}6H_2O$	24.7% Ni
Nickel Sulfate	$NiSO_4{\cdot}6H_2O$	22.3% Ni
Nickel Sulfamate	$Ni(NH_2SO_3)_2$	23.2% Ni
Nickel Carbonate	$NiCO_3$	46% Ni

2.3.1. Nickel Electroplating Process

As a basic electrodeposition process in nickel plating, soluble nickel-based salts dissolve with other constituents in an electrolyte bath. After dissolution is complete, the nickel-based salts decompose into positively charged nickel ions and negatively charged ions. When current applies, the positive ions that are reduced by two electrons become metallic nickel and accumulate on the cathodic surface. At the anodic side, metallic nickel dissolves to pos-itively charged ions in the bath that provide positive ions to complete the coating (Nickel Institute, 2014).

2.3.1.1. Rate of Deposition

Electroplating studies of Michael Faraday showed that there is a proportional ratio between coating metal amount on the cathode and dissolved amount from the anode by means of current and deposition time. Additively Faraday referred to that deposited metal amount or a dissolved amount related to their atomic weight and electron numbers which play a role in the electrochemical reaction and these electrons are called valency of the metal. The ratio between the atomic weight of metal and valency of the metal gives the chemical equivalent or equivalent weight of the metal.

Required electricity for deposited or dissolved metal can be found as 96,500 coulombs (ampere-seconds) for 1-gram equivalent of a metal which is constant. This is known as Faraday's constant.

Using these equivalences, the nickel deposited amount at the cathode or the amount of dissolved from the anode can be found from the following expression:

$$W = 1{,}095\ x\ I\ x\ t$$

where, W is nickel deposited amount at the cathode (or dissolved at the anode) in grams, I is the current that flows through the plating bath in amperes and t is the time that the current flows in hours. The factor 1.095 in the above calculation is derived from Faraday's work expression.

The equivalent weight is 29.35 which is calculated from the atomic weight of nickel is 58.70 divided by 2 which is the valency of nickel. This means that 29.35 grams of nickel or 1.095 grams will deposit or dissolve per 1 ampere-hour. This expression assumes that an efficient current does not dissipate, and efficiency is 100% in deposition and dissolution of metal. Therefore, at full current efficiency, 1.095 grams of nickel will deposit or dissolve (Nickel Institute, 2014).

2.3.1.2. Current Efficiency

The current efficiency of cathode is the percentage of the actual current for plating the nickel at the cathode to the total applied current. The current efficiency of cathode varies from 90% to 97% depends on types of nickel solution. In general, bright nickel solutions have lower cathode current efficiency and commonly nickel solutions have 95.5% of cathode current efficiency for special purposes. In order to increase the current efficiency of cathode, additive-free nickel solutions are used, and their efficiency is around 96%.

Similarly, the current efficiency of anode is the percentage of current that produces nickel ions at the anode and for dissolution of nickel, anode efficiency is always near the 100% when all operating parameters like pH and chloride content are provided in the range. The reason for the increased the nickel ion concentration and pH during plating is that the efficiency of anode is usually greater than cathode current efficiency. Anyhow, the system can be balanced the concentration of ions by leakage or drag-out.

Besides, current also is consumed by secondary electrochemical reactions. Discharging the hydrogen atoms from water cause a small amount of current consumption. This occurrence observes as a bubbles form at cathode surface (Nickel Institute, 2014).

2.3.1.3. Average Coating Thickness

The average coating thickness can be found from the relationship between the volume of the deposited part that is calculated from dividing the weight of the nickel deposited layer by the density of nickel and surface area of the deposited region. Also, the relation between current and time can be used as an alternative to calculate the average thickness.

$$T = \frac{12.294 \; x \; I \; x \; t}{A}$$

where T is the average thickness, A is the surface area of deposited region, I is the current and t is a time of current flows. This calculation comes from cathode current efficiency when it is 100%. It shows that average thickness depends on current density and time meantime weight of nickel deposited depends on current and time. Required time and current density can be used from table to be obtained desired average thickness value (Nickel Institute, 2014).

Table 3 - Current Density vs. Thickness

Time (min) to deposit the desired average thickness of nickel at various current densities.								
Current Density (Adm^{-2})	5 µm	10 µm	15 µm	20 µm	25 µm	30 µm	40 µm	50 µm
1	26	51	77	100	128	154	200	255
1.5	17	34	51	68	85	102	140	170
2	13	26	39	51	65	78	100	130
2.5	11	22	32	42	53	64	84	105
3	8.5	17	26	34	43	52	68	85
4	6.5	13	20	26	33	40	51	65
5	5	10	15	20	25	30	41	50
6	4.5	8.5	13	17	22	26	34	43

2.3.1.4. Thickness Distribution of Deposited Layer

Coating thickness can be varied at a different part of the substrate surface when the sample has a complex shape. The measured thickness from any point of the substrate is referred to as local thickness. The minimum value of the local thickness among all measured point considers estimating the service life or to achieve the requirements. Throwing power and current distribution play a role of the distribution of thickness (Nickel Institute, 2014).

2.3.1.5. Current Distribution

he true thickness of any point on the surface depends current density belong at that point. The current distribution depends on the geometrical shape of parts and their positions with respect to the anode position. The current density is higher the edges and the places which are close to anodes because the distance between anode and edges is short and that causes lower resistance to current flow. Increment of the resistance against to current flow causes the lower current so the coating thickness is lower at inside or shaded parts of the material. To avoid the inequality of coating thickness, arrange the position of parts, distance between anode and parts, using the shields or auxiliary anodes. Auxiliary anodes are used where the thickness is desired to be increased by placing to close the cathode. Shields, which are non-conductive material, are used between cathode and anode to monitor the current flow (Nickel Institute, 2014).

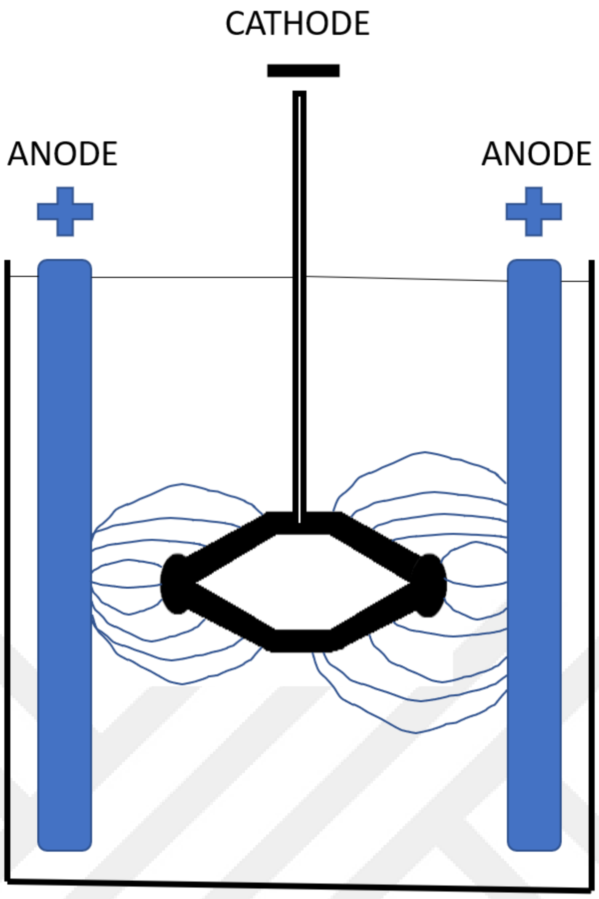

Figure 3 - Current Distribution

2.3.1.6. Throwing Power

Throwing power is a complex relationship between cathode polarization, the conductivity of electroplating solution, cathode efficiency and current density, and geometric factors. The throwing power increases with a decrease in the current density, increases the electrical conductivity of a solution, increases the distance between anode and cathode, increases the pH and temperature. Throwing power is determined experimentally and can take a positive or negative value. While the difference between the coating thickness at the top and the cove points decreases in positive solutions, the opposite situation can be obtained from negative solutions.

In nickel plating, generally throwing power value of solutions decreases in the positive region. Anyhow some bright nickel solutions have negative values (Nickel Institute, 2014).

Table 4 - Current Density and Throwing power for electrolytic solutions *(Di Bari)*

Solutions	**Average Current Density**	**Throwing Power (%) at Primary Current Density Ratios**		
	A.dm^{-2}	**5: 1**	**12: 1**	**25: 1**
Watts Nickel	4.3	8	7	14
Sulfamate Nickel	4.3	11	13	19
All-Chloride Nickel	4.3	18	18	27
Na/High Sulfate	4.3	23	31	40
Mg/High Sulfate	4.3	16	18	32
Proprietary Bright Nickel A	4.3	1	-12	-6
Proprietary Bright Nickel B	4.3	3	-12	-6
Acid Copper	4.3	0	-29	-61
Rochelle Copper	4.3	86	91	93
Conventional Chromium	16	-42	-48	-100

2.3.1.7. Internal Stress

While electrodeposition, some stresses can occur due to impurity as hydrogen and sulfur due to co-deposition or electro-crystallization process. If the occurred stress is tensile, cracks can occur on the coating layer, and the contrary coating layer tends to expand while compressive stress acts dominant inside the material. Internal stress plays an important role in adhesion between the coating layer and the substrate surface.

In nickel electrodeposition, stress has a wide range depending on the solution type and operational conditions. In watts nickel electrodeposition solution, tensile stress varies between 125 to 185 MPa, and at sulfamate nickel electrodeposition solutions have lower tensile stress value. Using sulfur with contain organic additives in nickel electrodeposition solution, compressive stress can be occurred (Nickel Institute, 2014).

2.3.1.8. Levelling and Micro-throwing Power

The substrate surface is not always flat. There are some defects or scratches on the substrate surface. Electroplating is filling these surface defects to have a perfect surface finish shows the leveling ability of the deposition process. In general, solutions whose purpose is decorative as bright nickel deposition solutions have a good leveling ability, and that property depends on current flow and current density.

Micro-throwing power is an ability to fill the small crevices without the leveling process. Sound and low porosity coating layer can obtained thanks to micro-throwing, and for this reason, additive-free nickel deposition solutions are preferred which have good micro-throwing properties (Nickel Institute, 2014).

2.3.1.9. Polarization

The surface of the electrode is polarized in the case of the applied voltage across the electrochemical bath during the deposition process. The difference of electrode potential from the potential of equilibrium is called polarization. In the deposition process, two types of polarization observe, one is called concentration polarization, and the other is called activation polarization. Polarization magnitude found as measured polarization that is called overpotential divided by electrode equilibrium potential. Total polarization potential at cathode described as,

$$\eta = \eta_a + \eta_c$$

where η is cathode potential, η_a is overpotential of activation polarization and η_c is overpotential of concentration polarization.

$$\eta_{\mathrm{a}} = \frac{R\,T}{n\,F} \ln \frac{i}{i_0}$$

$$\eta_c = \frac{R\,T}{n\,F} \ln \frac{i_d}{i_d}, \qquad i < i_d$$

where R is gas constant, T is absolute temperature, i is cathode current density, i_d is limiting current density, i_0 is exchange current density, n is valance of metal ions and F is Faraday constant (Zhu, Lei, Qu, & Xu, 2002).

2.3.1.10. Grain Size

It is well known that grain size and structure type affect the mechanical, chemical, and physical properties of the material. In nanocrystalline materials, a high density of boundaries and large volume fraction are observed due to very small grain size. There is a relationship between grain size and the overpotential of cathodic polarization. The crystallization process involves nucleation and growth. Fine-grain structure indicates that the nucleation process is dominant and activation polarization can increase the nucleation process (Zhu, Lei, Qu, & Xu, 2002).

2.3.1.11. Current Density

Current density has various effects on polarization. When the current density is increased, it leads to the increment of activation polarization and overpotential of concentration polarization. The concentration polarization overpotential directly affects the crystallization process. If the concentration polarization overpotential rises due to increment of current density especially it approaches the limiting current density value. The electroformed deposition has an unacceptable structure. Limiting current density depends on thickness, electrolyte concentration, and diffusion parameters and it is found as;

$$i_d = \frac{n\,F\,D\,c^0}{\delta}$$

where D is diffusion coefficient, c^0 is concentration of ion in electrolyte and δ is diffusion layer thickness (Zhu, Lei, Qu, & Xu, 2002).

2.3.1.12. Current Type

Pulse current affects the mass transportation in the bath solution. It also increases the limiting current density because of the metal ions during the replenishment time. If the frequency of pulse current is short pulse time and relatively long replenishment time, it provides growth of the nucleation process, and this results in finer structure for electroformed layer (Zhu, Lei, Qu, & Xu, 2002).

2.3.2. Types of Nickel Electroplating

2.3.2.1. Decorative Nickel Plating

Before organic additives were started to use in electrodeposition baths, between 1870 to 1945 decorative nickel plating was done by mechanic polishing. In the late 1940s, nickel electrodeposition began, and this development encouraged the multilayer nickel coating. In 1927, a thin chromium layer was plated on a mechanically polished nickel layer that prevents the yellowing or tarnishing. According to the development of this plating layer, bright nickel coating is existing today. Modern decorative nickel electrodeposition contains organic additives which innovate crystallization process that affect the brightness of appearance of layer and micro discontinuous chromium that helps protecting and enhance the appearance (Di Bari).

2.3.2.2. Engineering Plating

Nickel deposition for engineering purposes should be sulfur-free and matte appearance surface finish. Brightness is not a favored priority as in decorative applications. The important priority of engineering application of nickel plating is improving mechanical properties as corrosion and wear resistance and salvage or build-up worn or undersized parts and modifying magnetic properties for electronic applications. Engineering applications consist in the various industrial areas such as chemical, nuclear, telecommunications, consumer, electronics, and computer industries (Di Bari).

2.3.2.3. Electroforming

Electroforming and electrodeposition are different processes from each other with one major difference. As known, in the electrodeposition deposition layer and substrate surface are mechanically connected to each other. Indifference, electroforming coating metal is deposited on a mold and later coating separated from the mold. Electroforming process uses various application areas than electroplating as in the textile, aerospace, communication, electronics, automotive, photocopying, and entertainment industries (Di Bari).

Table 5 - Composition of electroforming solutions *(Sadiku-Agboola, Rotimi Sadiku, & Biotidara, 2012)*

Composition	***Sulfamate***	***Fluoborate***	***Watts***
Nickel Sulfamate (g/L)	328.667 – 493	-	262.933 – 361.533
Nickel Fluoborate (g/L)	-	328.667 – 493	-
Nickel Chloride (g/L)	0 – 16.433	-	41.083 – 57.517
Boric Acid (g/L)	32.867 – 49.300	24.650 – 41.083	32.867 – 49.300
Temperature (°C)	37,78 – 60	48.89 – 54.44	46.11 – 60
pH (electrometric)	3.5 – 4.5	3.0 – 4.0	1.5 – 4.0
Current Density (A/dm^2)	2.691 – 21.528	2.691 – 10.764	2.691 – 10.764
Tensile Strength (MPa)	413.686 – 758.423	386.107 – 551.581	379.212 – 448.159
Hardness (DPH)	140 – 250	130 – 250	140 – 160
Elongation, 2 in. (%)	5 – 30	10 – 32	20 – 30
Internal Stress, Tensile (MPa)	6.895 – 41.387	103.421 – 165.474	124.160 – 151.685

2.3.3. Solutions of Nickel electroplating

There are several nickel electroplating solutions and according to the usage field the solutions are prepared with using other salts and elements.

2.3.3.1. The Watt Solutions

The watt solutions are widely used among all nickel solutions. The watt solutions which are preferred for decorative aims were enhanced by Prof. Oliver P. Watts in 1916. The Watt solution mainly forms from the mixing of a certain amount of nickel sulfate, nickel chloride, and boric acid. Brighteners are added to the solution to gain a bright appearance and they are of various kind and chosen with respect to the intended brightness level (Kopeliovich, 2013).

2.3.3.2. All-Chloride Solutions

The aim of the chloride solution is to create a thick coating layer. All- chloride solutions run at low voltage, but it causes internal stress inside the coating layer (Kopeliovich, 2013).

2.3.3.3. Sulphate Chloride Solutions

Sulfate chloride solutions produce a lower internal stress coating layer by comparing the all-chloride solution deposition layer. It contains as same as the watt solution except for brighteners. It operates lower voltages and higher deposition rates than the watt solutions for the coating layer (Kopeliovich, 2013).

2.3.3.4. Fluoborate Solutions

They are using to create a thick deposited layer and for electroforming (Kopeliovich, 2013).

2.3.3.5. Nickel Sulfamate Solutions

First sulfamate plating solutions including nickel published in 1930 by R. Piontelli and L. Cambri. The first U.S. patent for sulfamate plating covering nickel belongs to M. E. Cupery in 1943. In 1950, Richard described nickel sulfamate coating, which has been used in the electrical industry since 1949 (Baudrand, 1996). After the development of technology and successfully made research the material properties of electroplated metals from nickel sulfamate solutions are also developed. Compering with other type of nickel electroplating methods electrodeposition from nickel sulfamate solutions is a low-cost method of nickel electroplating (P. Cojocaru, 2010). Also, sulfamate electrolytes seems more stable to pH changes comparing with sulfate and chloride electrolytes (Goldbach & Lapicque, 2000). Another advantages of nickel sulfamate bath based electroplating is that due to the its high solubility than chloride and sulfate baths, nickel sulfamate solutions are often preferred in the electroplating industry (Knapp, 1989). Moreover using the nickel sulfamate solution for nickel electroplating provides ductile behavior, although chlorides and Watts type nickel causes more brittle behavior (D. Golodnitsky & G.A.). Instead of this ductile behavior, the coating with sulfamate solution has the highest microhardness value comparing with other nickel electroplating solutions (Moharana & Mallik, 2013).

Table 6 – Solutions compositions and operational parameters (Di Bari)

	Electrolyte Composition (g/L)		
	Watts Nickel	**Nickel Sulfamate**	**Basic Semibright Bath**
Nickel Sulfate, $NiSO_4 \cdot 6H_2O$	225 - 400	-	300
Nickel Sulfamate, $Ni(SO_3NH_2)_2$	-	30 - 45	-
Nickel Chloride, $NiCl_2 \cdot 6H_2O$	30 - 60	300 - 450	35
Boric Acid, H_3BO_3	30 - 45	0 - 30	45
Operational Conditions			
Temperature (°C)	44 - 66	32 - 60	54
Agitation	Air or Mechanical	Air or Mechanical	Air or Mechanical
Cathode Current Density (A dm^{-2})	3 - 11	0.5 - 30	3 - 10
Anodes	Nickel	Nickel	Nickel
pH	2 – 4.5	3.5 – 5.0	3.5 – 4.5
Mechanical Properties			
Tensile Strength (MPa)	345 - 435	415 - 610	-
Elongation (%)	10 - 30	5 - 30	8 - 20
Vickers Hardness (100 g load)	130 - 200	170 - 230	300 - 400
Internal Stress, Tensile (MPa)	125 – 185	0 – 55	35 – 150

2.4. Composite Electroplating

Composite electroplating is a technological plating method to provide functional composite coating with using insoluble or soluble particles in electrolyte bath (Cai, ve diğerleri, 2010). the composite electroplating is preferred technique due to low cost and low temperature operation to produce proper metal matrix composite coating for multipurpose (Zhou, Zhang, & Qian, 2007). Metallic or non-metallic, polymeric fine particles are used to produce coating layer to improve the material properties as wear resistance, corrosion resistance or lubrication. Copper, chromium, nickel, iron, cobalt, gold, silver, nickel-boron and nickel-phosphorous are used as a metal matrix and metals, metal oxides, carbides, borides and polymers are used as fine particles. Molybdenum disulfide and polytetrafluoroethylene (Teflon) are used for lubricity while diamond and ceramic particles are used to increase wear resistance (Kim & Yoo, 1998). Composite deposition can be done in thermal spray, chemical vapor deposition, physical vapor deposition methods than electroplating (Gül, et al., 2011). Low cost, high production rate, maintenance easily and working easily at low temperatures, continuous processing ability, deposited at normal pressure, reduced waste, homogenously distribution of fine particles and complex geometry capability are reasons that electroplating production method stands out among these techniques (Rostami, Fahami, Nasiri-Tabrizi, & Ebrahimi-Kahrizsangi, 2013).

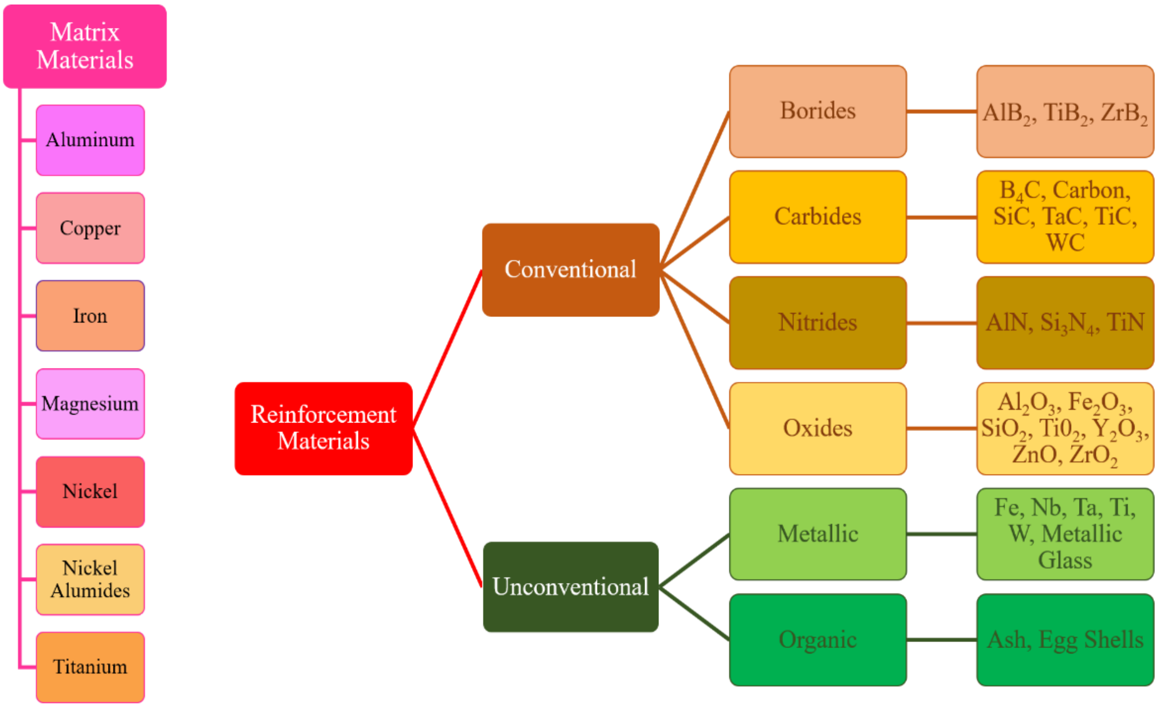

Figure 4 - Content of Composite Material *(Seetharaman & Gupta, 2021)*

2.4.1. Metal Matrix

The mechanical properties such as hardness, wear resistance and performance against the corrosion of the material are increased with the metal matrix composite electroplating (Özkan, Hapçı, Orhan, & Kazmanlı, 2013).

The metal matrix is soft material which has excellent mechanical and physical properties. Aluminum, magnesium, copper, and titanium are preferable soft metal matrix because of good ductility, thermal and electrical conductivities, malleability, and machinability properties (Boostani, et al., 2015).

Increasing these mechanical properties depends on the matrix material, its quantity, and its phase also the size and distribution of the fine particles used. Besides, the operational conditions of composite electroplating like temperature, agitation speed, bath composition and current density affect the properties of matrix phase (Özkan, Hapçı, Orhan, & Kazmanlı, 2013).

In composite materials, matrix material has two main functions. First of all, matrix material holds in balance the fine particles which is called reinforced elements. Secondly, matrix material cannot lean the force or load, so matrix connects with reinforced elements and divide the load among all reinforced elements equally. As a conclusion, metal matrix composite materials show stability to preserve the failure of material due to fracture or crack (Selvam, Dinaharan, & Rai, 2021). The important thing in the selection of matrix material and reinforced material is that the matrix material and the reinforced material do not react chemically. Otherwise, matrix material will damage the reinforced material, so composite material will lose the stability and strength (Selvam, Dinaharan, & Rai, 2021).

Table 7 – Metal Matrix Materials' Properties *(Seetharaman & Gupta, 2021)*

Base Matrix Alloys	**Density (g/cc)**	**Modulus (GPa)**	**Yield Strength (MPa)**	**Ductility**
Magnesium	1.74 – 1.95	42 - 47	70 – 400	3.5 – 18
Aluminum	2.5 – 2.9	68 – 82	30 – 500	1 – 44
Titanium	4.4 – 4.8	90 – 120	250 – 1250	1 – 40
Steels	7.1 – 8.0	187 – 215	240 – 690	18 – 31
Nickel	7.75 – 8.65	150 – 245	300 – 1900	0.5 – 60
Copper	8.93 – 8.94	112 - 148	300 - 500	3 - 50

2.4.1.1. Aluminum Alloy Metal Matrix

After steel, aluminum most used metal in industry due to easily available. Besides being ductile and soft and having good conductivity and corrosion resistance, aluminum provides strength and lightness with alloying elements (Mondolfo, 1976).

Table 8 – Properties of aluminum and its alloy *(Rajak & Menezes, 2021)*

Aluminum Alloys Series	**Composition**	**Yield Strength (MPa)**	**Tensile Strength (MPa)**	**Elongation (%)**	**Vickers Hardness (HV)**	**Usage Area**
1XXX	Al	70 - 150	90 - 165	10 - 40	25 - 44	Pure Aluminum
2XXX	Al - Cu	90 - 330	170 - 420	16 - 24	80 - 115	Aviation Sector
3XXX	Al - Mn	60 - 115	90 - 155	10 - 26	30 - 45	Canning Industry
4XXX	Al - Si	70 - 180	110 - 250	8 - 20	30 - 55	Welding rods and Brazing sheet
5XXX	Al - Mg	60 - 260	170 - 330	15 - 35	60 - 95	Structural and Architectural purposes
6XXX	Al - Mg - Si	55 - 290	100 - 370	10 - 30	60 - 105	Automobile Sector
7XXX	Al - Zn - Cu	170 - 390	230 - 575	3 - 10	50 – 165	Aviation Sector

Aluminum metal matrix composites have several properties which are:

i. High strength and high stiffness
ii. Reduced density and controlled weight
iii. Improved creep resistance at high temperature, electrical conductivity, and wear resistance
iv. Moderate thermal expansion coefficient, toughness, and ductility

In addition, several reinforcement materials can be used in aluminum metal matrix composites (Rajak & Menezes, 2021). They are silicon carbide reinforced, aluminum oxide reinforced, boron carbide reinforced, fiber reinforced, zircon reinforced, and fly ash reinforced (Moonaa, Waliab, & Rastogib, 2018).

2.4.1.2. Magnesium Alloy Metal Matrix

Magnesium which is softest metal in industry has a hexagonally closed pack structure with 1.7 g/cm3 density. Magnesium is lower conductive thermally than aluminum, but thermal expansion coefficient is so close to aluminum. In the industry, alloying elements are added to magnesium to increase the properties. Especially the main problem of magnesium is corrosion resistance. From auto components to sports products, magnesium and its alloys are used in several industry areas since it has good machinability and is light and easy to cast (Avedesian & Baker, 1999).

Table 9 – Properties of magnesium and its alloys *(Rajak & Menezes, 2021)*

Magnesium Alloy Series	**Density (g/cm3)**	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Elongation (%)**	**Vickers Hardness (HV)**
AZ91	1.81	240	165	3	70
AM60	1.80	220	130	8	65
AS41	1.77	215	140	6	55
AE41	1.82	245	142	10	62
AJ62	1.8	235	140	10	62
ZM21	1.8	200	124	9	55
AZ80	1.8	330	230	12	70

Magnesium metal matrix composite materials have various properties which were listed below.

i. Light weight
ii. Excellent stiffness to weight ratio
iii. Low density (%33 less than aluminum)
iv. Excellent mechanical properties at high temperature
v. Good corrosion resistance
vi. High specific strength

vii. Improved damping
viii. Low thermal expansion coefficient

There are some magnesium based metal matrix composite material which are silicon carbide reinforced magnesium (SCRM), boron carbide reinforced magnesium (BCRM), fiber reinforced magnesium (FRM), CNT reinforced magnesium (CNT-RM), aluminum oxide reinforced magnesium (AORM), titanium carbide reinforced magnesium (TCRM) (Rashad, Pan, & Asif, 2015).

2.4.1.3. Copper and Alloys Metal Matrix

Copper which is earliest discovered metal is used in electrical and high temperature applications since copper has good thermal conductivity and electrical conductivity in addition to this copper has good corrosion resistance and face centered cubic structure (Davis, 2001).

Table 10 – Properties of copper and its alloys *(Rajak & Menezes, 2021)*

Copper Alloys Series	**Composition**	**Property**	**Usage Area**
C1XXX	Cu	Pure Copper	Electrical Components
C2XXX	Cu - Zn	High Strength	Automobile and Defense Sectors
C3XXX	Cu – Zn – Pb	Good for Cold	Electrical Components
C4XXX	Cu – Zn – Sn	Good Corrosion Resistance	
C5XXX	Cu – Sn	High Fatigue and Strength	Springs
C6XXX	Cu – Al		Automotive and Ship building industries
C7XXX	Cu – Ni	Good Thermal Conductivity	High Temperature Application

i. Low thermal expansion coefficient
ii. High elastic modulus
iii. Good electrical conductivity and wear resistance
iv. High thermal conductivity

are the properties of copper metal matrix composite materials (Rajak & Menezes, 2021).

Table 11 – Properties of copper metal Matrix Composite Material

Property	**Values**
Density (g/cm3)	2.85
Elongation at break (%)	6
Modulus of tensile (GPa)	100
Longitudinal tensile strength (MPa)	610
Volume fraction of SiC (%)	17
Yield Strength (MPa)	400

2.4.1.4. Titanium and Alloys Metal Matrix

Titanium, which has a low-density metal, has two different crystal structures. At room temperature, titanium has hexagonal closed packed (HCP) crystal structure which is called alpha, α, titanium while at 883℃ titanium has body-centered cubic (BCC) crystal structure which is called beta, β, titanium because of phase transformation from alpha to beta phase. These two phases have suitable properties for different applications thanks to the different alloying elements which they contain. Generally, titanium is used in biomaterials, marine and petrochemical industries due to its corrosion resistance, biocompatibility, and good strength (Bache, 2003).

Titanium metal matrix materials mainly divided in two groups based on the reinforced material’s form which are continuously reinforced titanium metal matrix composites and discontinuously reinforced metal matrix composites (Welsch, Boyer, & Collings, 1993).

Table 12 – Properties of titanium and its alloys *(Rajak & Menezes, 2021)*

Property	**Conventional Titanium Metal Matrix Composite**	**Titanium Aluminide Reinforced Titanium Metal Matrix Composite**	**Super-alloys**
Density (g/cm^3)	4.04	4.18	8.3
0° Stiffness (GPa)	200	242	207
90° Stiffness (GPa)	145	200	207
Maximum Useable Temperature (°C)	538	760	1090
0° CTE (°C^{-1}/10^6)	7.20	7.92	13.0
90° CTE (°C^{-1}/10^6)	8.91	9.18	13.0

2.4.1.5. Iron and Alloys Metal Matrix

Iron which is in the earth's crust is difficult to use in industry because pure state of iron has high melting point to get shape. Different alloying elements add different properties to iron and allow it to be used in industry. The best examples of these are cast iron and steel which have different carbon content. Instead of iron, cast iron and steel, which are an alloy of iron, are widely used in the industry for their properties. Cast iron, which is obtained from iron, which is excessive as raw material, has a usage area of 95% in the world due to its strength and the ability to easy shape. The steels that can adapt to the desired properties with the amount of carbon they contain, especially the stainless-steel group which contains chromium, have a widespread use in the industry thanks to their resistance to corrosion (Mbuya, 2003).

2.4.1.6. Nickel and Alloys Metal Matrix

Nickel has FCC structure, and it is a good ductile metal. Nickel is always an important metal for many industries, since nickel allows them to be used in extremely high-performance applications as it is a highly flexible metal that is probably almost entirely alloyed with other metals. Nickel and its alloys are excellent corrosion resistance, high density, and excellent magnetic and electronic properties. Nickel-chrome alloy array and nickel-chromium-iron increased strength and was used in high temperature applications (Thompson, 2001).

2.4.2. Reinforcements

Composite electroplating is done on the surface since increase the mechanical properties of surface (Rostami, Fahami, Nasiri-Tabrizi, & Ebrahimi-Kahrizsangi, 2013). Reinforced element which is called reinforcement strengthens the matrix material in composite material and has a good bonding ability. Reinforced elements are harder and lighter than matrix material which bring strength and stiffness with respect to weight to material (Dinaharan, Murugan, & Parameswaran, 2011).

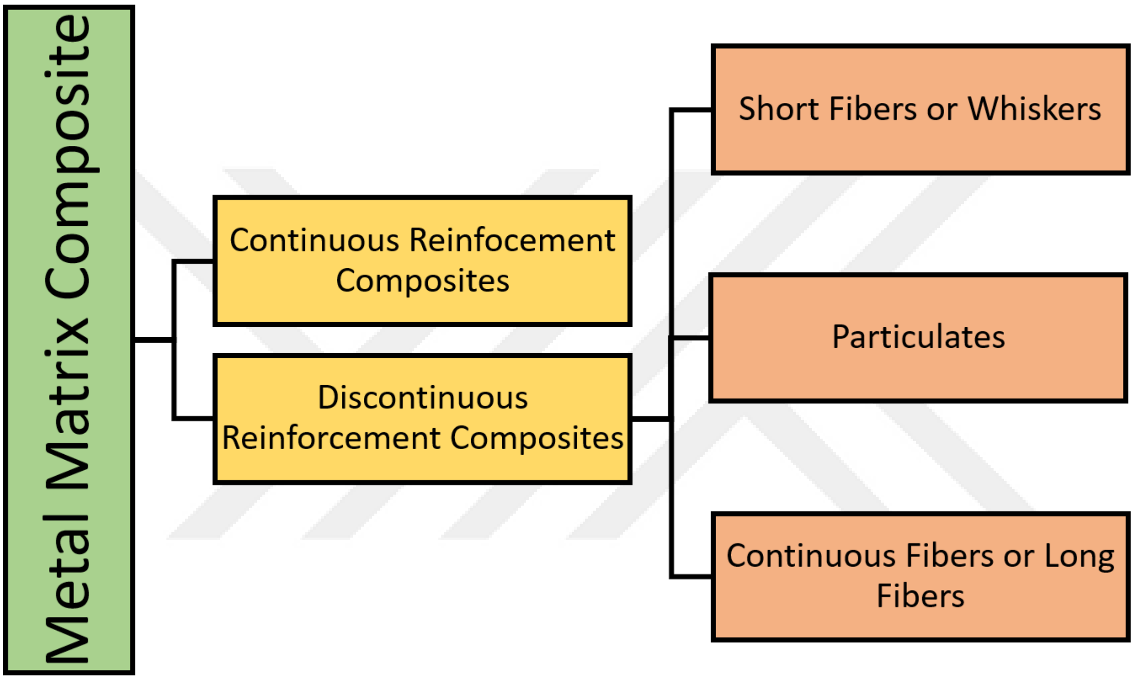

Figure 5 - Types of reinforcements *(Rajak & Menezes, 2021)*

During the composite electroplating, apart from the use of metals with high mechanical properties, nanoparticles are used in coating to give more durability to the coating against the environmental damages such us wear or corrosion (Rostami, Fahami, Nasiri-Tabrizi, & Ebrahimi-Kahrizsangi, 2013).

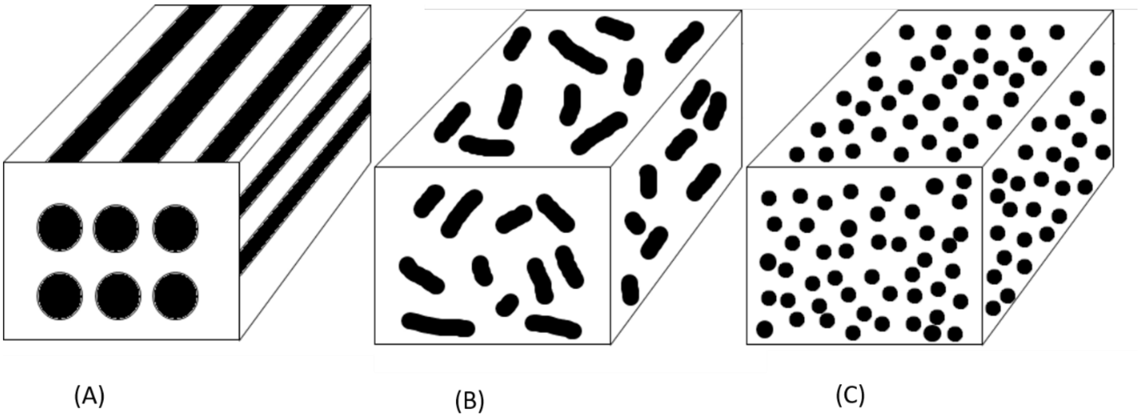

Figure 6 - Types of Reinforcement A)Long fiber reinforcement, B)Short fiber reinforcement, C)Particle reinforcement *(Rajak & Menezes, 2021)*

Table 13 – Some reinforcement materials and their properties

Reinforcement Material	**Density (kg/m^3)**	**Meting Point (°K)**	**Thermal Conductivity (W/m°K)**	**Thermal Coefficient Expansion (x10^6/°K)**	**Vickers Hardness (HV)**	**Young's Modulus (GPa)**
SiC	3200	3073	270	5.1 – 5.8	2800	560
B_4C	2520	2780	32.5	7.3	1855	472
Al_2O_3	3855	2323	29	8.5	1780	460
TiC	4770	3413	31.8	7.6	2270	450

2.4.2.1. Silicon Carbides (SiC) Nanoparticles

As a non-oxidizing ceramic, silicon carbide (SiC) has a significant place in the industry due to its suitable properties as high hardness, strength, high melting point, oxidation and high erosion resistance and it is stable chemically and thermally. These properties allow it to be preferred in abrasion and cutting applications and to be used at high power and high temperature electronic devices. In 1892, silicon carbide produced by Acheson. Although the production method is multi-stage and complicated, low quality product was obtained by this method. Higher quality production was started with the advanced of technology. These production methods are physical vapour deposition, chemical vapor deposition, sol-gel, liquid phase sintering and mechanical alloying (Abderrazak & Hmida, 2011).

Table 14 – Properties of Silicon Carbide *(AZoNano, 2013)*

Chemical Symbol	**SiC**
Group	Silicon 14
	Carbon 14
Electronic Configuration	Silicon [Ne]$3s^2 3p^2$
	Carbon [He]$2s^2 2p^2$
Chemical Composition Content (%)	Silicon: 70
	Carbon: 29.94
Density	3.22 g/cm^3
Molar Mass	40.11 g/mol
Melting Point	2730°C
Apperence	Grayish white powder
Applications	·As a high-grade refractory material, special material for polishing abrasive, various ceramic parts, textile ceramics and high frequency ceramics ·Manufacture of rubber tires ·Manufacture of grinding material having a high hardness ·Making of sealing valves that withstand high temperatures ·Resistance heating element manufacture ·Used in modifying the strength of alloys ·High temperature spray nozzle manufacture ·Substrates for ICs ·Mirror coatings for high ultraviolet environments

Polytypes are silicon crystalline carbide forms and they have more than 250 different forms. 3C, 4H, 6H and 15R are most accustomed polytypes (Abderrazak & Hmida, 2011).

Table 15 - The main physical/chemical properties of the most widespread SiC polytypes *(Andrievski, 2009)*

PROPERTY	**POLYTYPE**			
	3C – SiC (β-SiC)	2H – SiC (α-SiC)	4H – SiC (α-SiC)	6H – SiC (α-SiC)
Space Group	F43m	P6$_3$mc	P6$_3$mc	P6$_3$mc
Hexagonality (%)	0	100	50	33
Stacking Order	ABC	AB	ABCB	ABCABC
Lattice Constant a (nm)	0.43589 – 0.43596	0.30753 – 0.3081	0.3070 – 0.3081	0.3073 – 0.3081
Lattice Constant c (nm)	-	0.5031-0.5048	1.0053 – 1.008	1.51092 – 1.512
Density (g/cm3)	3.215	3.219	3.215	3.212
Band Gap (eV)	2.39	3.33	3.26	3.0
Thermal Conductivity (Wcm^{-1} K^{-1})	3.6	-	4.9	4.9
Electron Mobility (cm^{2}V^{-1}S^{-1}, 300K)	≤ 1000	-	≤ 850	≤ 450
Hole Mobility (cm^2 V^{-1}s^{-1}, 300K)	≤ 40	-	≤ 120	≤ 100
Electrical Resistivity (Ωcm)		10^2 – 10^3		

2.4.3. Production Methods of Metal Matrix Composites

Metal matrix composite materials are produced in a several ways and they are listed below:

1) Liquid State Processing
    a) Melt Deposition
        i) Immersion plating
        ii) Electroplating
        iii) Chemical vapor deposition
        iv) Physical vapor deposition
        v) Spray deposition
        vi) Disintegrated melt deposition
    b) Stir Casting
    c) Melt Filtration
        i) Pressure die infiltration
        ii) Gas pressure infiltration
        iii) Ultrasonic infiltration
        iv) Centrifugal infiltration
        v) Lorentz force infiltration
        vi) Squeeze casting
2) Solid State Processing
    a) Powder Consolidation
    b) Mechanical Alloying
        i) Post-processing (Sintering of powder)
        ii) Microwave assisted rapid sintering
    c) Diffusion Bonding
    d) Deformation Process
3) In-Situ Processes
4) Additive Manufacturing

In this study, only electroplating production method was examined. In electroplating, metal matrix composite material is produced from electrolytic solution which contain ions with applying current and all electroplating considerations are valid. Therefore, some limited alloy metal matrixes can be used in electroplating method. Main advantages of electroplating method for metal matrix composite materials is that reinforced material is escaped damages.

Nevertheless, some mechanical problems can be occurred such as adhesive and porosity (Seetharaman & Gupta, 2021).

2.4.4. Properties of Metal Matrix Composite Electroplating

The properties of the metal matrix composite coating depend on the properties of the metal matrix and the reinforced material which is contains. Metal matrix and reinforced material show different behaviors and properties by being affected by different conditions. The composite material is also affected by the interface between matrix material and reinforced material (Seetharaman & Gupta, 2021).

2.4.4.1. Volume and Weight Fraction

The property of composite material depends on the mixture of volume ratio of metal matrix material and reinforced material. Hereby, relative proportions of metal matrix material and relative material should know to determine the mechanical properties of composite material. The relative proportion can be calculated in terms of volume fraction of weight or mass fraction. Volume fraction is used for estimating of the properties while weight fraction is used for easy calculation of analytical balance. Therefore, conversion between in these two terms should know well. This relationship can be shown as:

$$W_m = \frac{\rho_m}{\rho_c} V_m$$

$$W_r = \frac{\rho_r}{\rho_c} V_r$$

where, W_m is weight fraction of metal matrix material, W_r is weight fraction of reinforced material, V_m is volume fraction of metal matrix material, V_r is volume fraction of reinforced material. ρ_c, ρ_m, and ρ_r are density of composite material, density of metal matrix material and density of reinforced material, respectively (Seetharaman & Gupta, 2021).

2.4.4.2. Density

Density can be calculated according to mixing rule giving by (Seetharaman & Gupta, 2021):

$$\rho_c = \rho_m V_m + \rho_r V_r$$

$$\rho_c = \frac{1}{\frac{W_m}{\rho_m} + \frac{W_r}{\rho_r}}$$

2.4.4.3. Thermal Expansion Coefficient

According to considerations of mixing rule, thermal expansion coefficient of composite material can be found as:

$$\alpha_c = \alpha_m V_m + \alpha_r V_r$$

where, α_c is thermal expansion coefficient of composite material, α_m is thermal expansion coefficient of metal matrix material, α_r is thermal expansion coefficient of reinforced material.

In continuous fiber reinforced metal matrix composite material, thermal expansion coefficient is calculated different with respect to longitudinal and transverse direction.

$$\alpha_{c,long.} = \frac{\alpha_m V_m E_m + \alpha_f V_r E_r}{V_m E_m + V_r E_r}$$

$$\alpha_{r,trans.} = (1 + V_m)\alpha_m + \alpha_f V_r$$

where, E_m is the elastic modulus of matrix material, E_r is elastic modulus of fiber reinforced material and v_m is the poison's ratio of matrix material.

For particle reinforced metal matrix composite materials, thermal expansion coefficient estimates as:

$$\alpha_c = \frac{(\alpha_m V_m K_m + \alpha_r V_r K_r)}{(V_m K_m + V_r K_r)}$$

where, K_m and K_r are bulk modulus of metal matrix material and bulk modulus of reinforced material, respectively (Seetharaman & Gupta, 2021).

2.4.4.4. Elastic Modulus

The volume fraction and kind of reinforcement material determine the modulus of elasticity of component material. Elastic modulus of composite material increases with increasing the amount of reinforced material and the modulus is estimated as:

$$E_c = E_m V_m + E_r V_r$$

For transverse direction,

$$E_{c,trans.} = \frac{1}{(\frac{V_m}{E_m} + \frac{V_r}{E_r})}$$

For discontinuous fibers and particles reinforced composite materials,

$$E_c = \frac{E_m(1 + 2sqV_m)}{1 - qV_r}$$

$$q = \frac{\left(\frac{E_r}{E_m} - 1\right)}{\left(\frac{E_r}{E_m} + 2s\right)}$$

where, s is aspect ratio (Seetharaman & Gupta, 2021).

2.4.4.5. Tensile Strength

Reinforced materials bring strength to the material. Therefore, metal matrix composite materials which has metal matrix material strength and reinforced material strength show more durability than metal matrix materials which is free reinforced material. tensile strength of metal matrix composite material given by:

$$\sigma_c = \sigma_m + \sqrt{\sum \Delta\sigma_i{}^2}$$

where, σ_c and σ_m are the strength of composite materials and the strength of metal matrix material, respectively. Δσ indicates strength contribution from different strength mechanism which are mentioned in below (Seetharaman & Gupta, 2021):

2.4.4.5.1. Transfer of Load

In metal matrix composite material, load is distributed to reinforcement materials by metal matrix material.

$$\Delta\sigma_{LT} = V_r \sigma_m \frac{(L + t)AL}{4L}$$

where, σ_m is yield strength of metal matrix material, t is thickness of reinforced material and L is length of reinforced material, A is the factor of geometry (Seetharaman & Gupta, 2021).

2.4.4.5.2. Thermal Residual Stress

Thermal cycling processes at high temperatures cause the residual stress. Thermal residual stress determines as:

$$\Delta\sigma_{TS} = \frac{E_r E_m}{E_r V_r + E_m V_m} V_r \Delta\alpha \Delta T$$

Where, Δα is mismatch of thermal expansion coefficient, ΔT is temperature difference (Seetharaman & Gupta, 2021).

2.4.4.5.3. Orowon Strength Mechanism

Fiber or particle reinforced materials limit the dislocation movement and the Orowon mechanism explains the strength mechanism of dispersion strengthened materials depends on strength and size and found as:

$$\Delta\sigma_{OR} = \frac{0.13bG}{d_r\left[\left(\frac{1}{2V_r}\right)^{1/3} - 1\right]} \ln\frac{d}{2b}$$

where, G is the shear modulus of metal matrix material, d is a size of reinforced materials and b is Burger vector (Seetharaman & Gupta, 2021).

2.4.4.5.4. CTE and EM Mismatch

In composite materials, dislocation density of matrix material higher than reinforced material because of the mismatch of the coefficient of thermal expansion (CTE) and modulus (EM) in between matrix material and reinforced material (Seetharaman & Gupta, 2021).

$$\Delta\sigma_{CTE} = \frac{A\Delta\alpha\Delta T V_r}{bdV_m}$$

$$\Delta\sigma_{EM} = \frac{6V_r}{\pi d}$$

2.4.4.5.5. Grain Refinement Strength Mechanism

In polycrystalline metals, grain boundaries prevent dislocation movement and increase the stress required for deformation. Comparing the matrix grain size of metal matrix composite materials which contain reinforced material with the grain size of metal matrix composite materials without reinforced material, metal matrix composite materials with contain reinforced material have a smaller grain size than those without reinforced material. This situation increases the yield strength of metal matrix composite materials and prevents the movement of dislocations with continuously in the slip plane. The yield strength of material is estimated by Hall-Petch equation:

$$\Delta\sigma_{HP} = \frac{k_y}{\sqrt{d}}$$

where, d is average grain size and k_y is characteristic constant of strengthening coefficient which is depends on material.

In studies on different strength mechanisms, the formulas which belong Orowon mechanism, coefficient of thermal expansion mismatch mechanism and load-bearing effect have been simplified (Seetharaman & Gupta, 2021):

$$\sigma_c = (1 + 0.5V_r)\left(\sigma_m + A + B + \frac{AB}{\sigma_m}\right)$$

$$A = 1.25G_m \sqrt[b]{\frac{12\Delta\alpha\Delta T V_r}{bdV_m}}$$

$$B = \frac{0.13G_m b}{d\sqrt[3]{\frac{1}{2}V_r - 1}} \ln\frac{d}{2b}$$

2.4.5. Nickel Matrix Composite Electroplating with SiC Nanoparticles

Silicon carbide nanoparticle in the nickel matrix composite electroplating has main two effect on crystallization of coating layer. In crystallization process which is consists of nucleation and growth processes, silicon carbide influence the nucleation process and it cause new nucleus. Besides nickel growth mechanism is affected from silicon carbide and the silicon carbide nanoparticles block the growth of nickel metals. This phenomenon is concluded with small grain structure (Özkan, Hapçı, Orhan, & Kazmanlı, 2013).

In the literature when silicon carbide content increase in the electrolytic bath solution, corrosion resistance of coating layer increase. While composite electroplating process is running, insoluble fine particles get involved in electrolyte bath solution. These insoluble fine particles which are distributed homogenously in the solution are captured in the coating layer (Gül, et al., 2011).

3. MATERIALS AND METHODS

3.1. Materials and Characterization

3.1.1. Cutting, Molding, Polishing and Grinding

3.1.1.1. Cutting

A good cutting process is crucial to the efficiency and function of quality control. Cutting machines and parts are designed to cause minimal damage to material and cut any size material. The compressed material is carefully and slowly cut from the desired location with the help of a manual arm or a hydraulic system by the operator. During this process, oil is often used to reduce the heat caused by friction between the material and cutting panels.

3.1.1.2. Molding

For surface treatments or testing, materials that have small dimensions are coated to increase dimensions. The most important detail in molding process is that the material to be used for molding does not react with the material to be coated. Usually powder or liquid polymer materials are used for molding process and there are two methods which are cold molding and hot molding. In cold molding process, liquid polymer mixture is poured into mold then, liquid polymer covers the material in the mold and is left to freeze. In order to avoid any problem in the next test stages, it should be left to freeze without any air bubbles after pouring into the mold. Only disadvantage of cold molding is that takes long times such as 24 hours to become solid. In hot molding, solid polymer particles pour into the cavity of machine and covers the material. during the process, molding machine heats the cavity therefore, solid polymeric particles melts while pressing. In this process, the material to be molded should have a higher melting point than solid polymer particles and should not react under heat.

3.1.1.3. Grinding

The grinding method is a surface preparation and cleaning method used for smoothing the material surface. It is applied to prevent erroneous measurements due to height differences in thin material under light. The material is grinded on the rotating benches of the grinding machine, starting from high particle size grinding paper to small particle size grinding papers. At the beginning, the material is held at an angle of 90° to the rotation direction, and the material is rotated 45° at each paper change. Besides, during the process water droplets are dropped to prevent the any failing due to the friction force.

3.1.1.4. Polishing

After the grinding process, polishing is applied to remove the tiny scratches remaining on the surface and to polish the surface. In the polishing machine, which has the same bench and operation as the grinding machine, a diluted solution which contain nano-sized ceramic particle such as diamond oxide or aluminum oxide is dropped on the soft-bristled polishing carpet to smooth the surface with these ceramic particles. In the same way, material is held with an 90° angle to the rotation direction and during the process water droplets are dropped to prevent the any failing due to the friction force.

3.1.2. Hardness Test

Hardness is the ability of materials to resist plastic deformation. The hardness test is a mechanical test due to its easy applicability and little damage to the material. A relationship can be established between the results obtained from the hardness test and the mechanical properties of the material. Since the resistance of materials to resist plastic deformation is related to the yield strength, the hardness can be determined relatively by applying such a force.

Hardness measurement is made by measuring the response of a tip which has different shapes and harder than the test material by pressing against the surface of the material with a known amount of force. There are points to be considered when performing the hardness test. Firstly, tip of the hardness machine material and test specimen surface should be parallel to each other. Secondly, thickness of tip material should be at least 10 times higher than tip area and third consideration is that the tip should not measure at the edges of the test material. When making sequential and continuous measurements on the surface of the same test material, each measurement point should be at least three times the tip diameter from each other. There are 4 hardness measurement test methods depends on tip material and shape which are Brinell hardness test, Rockwell hardness test, Vickers hardness test and micro hardness test.

3.1.2.1. Vickers Hardness Test

The Vickers hardness test, which is performed by pressing the 136° square pyramid diamond tip by applying a force to the material, is a test that measures the hardness by measuring the diagonals of the trace on the surface of the material. Vickers hardness test does not depend on force so the test can apply so soft and so hard materials. The Vickers hardness is calculated as:

$$HV = 1.8544 \times \frac{F}{d^2}$$

where, F is applied force and d is the diagonals of the trace on the surface of the test material (Karadeniz Technical University Lecture Note).

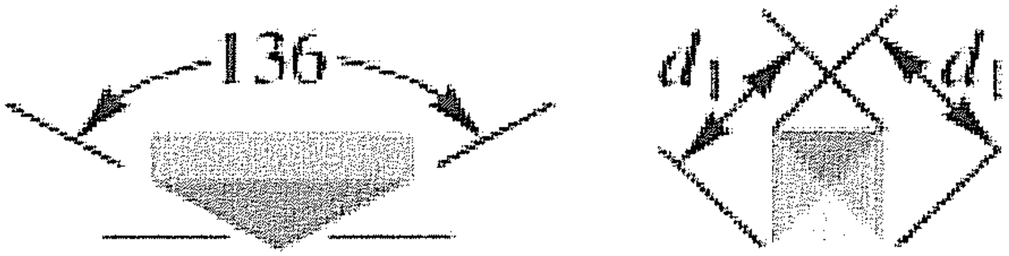

Figure 7 - Tip of Vickers Hardness Test

3.1.3. Optic Microscopy

The optical microscope, light microscope, which is the most necessary examination method of metallography, is a device that provides visualization of the structure of the materials by working based on the principle of light refraction. It provides a contrasting image by collecting the lights reflecting from the surface of the material with its lenses which have different magnifications. Magnification ratio of the microscope is found as:

$$M = \frac{v - f}{f}$$

where, v is the distance between the image and the lens and f is focal length of lens.

Most of the case, more than one lenses are used to magnification. In this case magnification ratio is obtained by multiplying the magnification of the lenses:

$$M = M_1 M_2 = \frac{(v_1 - f_1)(v_2 - f_2)}{f_1 f_2}$$

Resolution can be determined as:

$$R = \frac{d}{2} = \frac{0.61\lambda}{\mu \sin \alpha}$$

where, d is airy disc distance, μ is the refractive index of the environment between material and lens, α is half angle of the cone of light coming into the objective lens and μsinα refers to numerical clearance. For obtaining high resolution, maximum numerical clearance which is 1.5 and visible light that have short wavelength which is purple light that the wavelength is 400nm then maximum resolution is 162.6 nm for optic microscope.

Depth of field can be calculated as:

$$D_f = \frac{d}{\tan\alpha} = \frac{2R}{\tan\alpha} = \frac{1.22\lambda}{\mu \sin\alpha \tan\alpha}$$

There is an opposite relationship between resolution and depth of field. As the resolution increases, the depth of field decreases, so the material should be brought closer to the microscope lens for clear images since clear images are obtained only from objects within the depth of field (Bartın University Lecture Note).

3.1.4. Atomic Force Microscopy (AFM)

It provides high resolution, three-dimensional visualization of the surface with the help of a needle tip pointed to atomic dimensions. Imaging is performed by examining the interaction of the needle tip with the surface. Different needle tips are used for different purposes. Atomic force microscope can be used with three different techniques. These; The contact method, in which the needle is applied by contacting the surface, is the non-contact method where the needle does not touch the surface and the percussion method where the needle is applied by hitting the surface. In addition to imaging the sample surfaces, phase, electrical conductivity, and magnetic differences can also be detected. AFM is used for surface investigations of thin film coatings, surface investigation of organic and inorganic materials and surface examinations can be made on surface smoothness, phase differences, electrical conductivity differences and magnetic field direction differences.

3.2. Experimental Methodology

The literature about the nickel electrodepositions are reviewed. As a consequence of literature reviews, studies are mainly focused on processing of nickel deposition from nickel sulfamate solution bath with included silicon carbide (SiC) nanoparticles to improve corrosion and wear resistance and effect of SiC nanoparticles on nucleation and microstructure. The effect of SiC nanoparticles on corrosion and wear resistance are examined with different amount of SiC nanoparticles.

3.2.1. Solution Preparation

For production, nickel sulfamate salt ($NiSO_4.6H_2O$), boric acid (H_3BO_3), and silicon carbide (SiC) nanoparticles with (100nm) particle size are used. General nickel sulfamate solution contains 450g/L of nickel sulfamate salt and 30g/L of boric acid and deposition process runs at 50 c and 4 ph as optimum operational conditions. To compare the wear and corrosion resistance, one nickel sulfamate solution without the nanoparticles and six nickel sulfamate solutions with contain different amount of SiC nanoparticles are prepared.

Table 16 – Solutions for Set – 1

Sample Label	**Content of Nickel Sulfamate Salt**	**Content of Boric Acid**	**Content of SiC nanoparticles**	**pH**
S1	90.1	6	No Particle	3.95
S2	90.3	5.99	212.4	3.97
S3	90.7	6.06	30	3.99
S4	90.11	6.03	107	3.97
S6	90.15	6.14	2.3	4.06
S8	90.15	6.14	12	4.06

Table 17 - Samples Labels with SiC nanoparticle contents

	Content of Silicon Carbide Nanoparticles					
	No Particle	0.1 g/L	0.5 g/L	1 g/L	0.01 g/L	0.05 g/L
Set – 1	S1	S3	S4	S2	S6	S8
Set – 2	S7	S5	S10	S9	-	-
Set – 3	S11	S12	S14	S13	-	-

To prepare the solutions, grams of elements are calculated by using precision scales according to capacity of deposition bath at laboratory which is 200ml beaker with general nickel sulfamate solution composition. After all powders measured, they are mixed in 200ml baker with mechanic agitation at the same time solutions are heated until 50℃ and during the procedure it is kept constant. Solution pH should be 4 which is optimum value and to setup the pH KOH solution is added by droplet. Six 200ml nickel sulfamate solution is prepared and different amount of SiC nanoparticle is added according to 1g/L (S2), 0.1g/L (S3), 0.5g/L (S4), 0.01g/L

(S6), 0.05g/L (S8), respectively. It took approximately 1 hour for all the powders to melt and the solutions to become homogeneous.

3.2.2. Cathode Sample Preparation

Samples were copper plates which are cut rectangular shape and surfaces are cleaned with ethylene and then covered with opening one side area that is 2x2cm^2 by using kapton tapes which are hinder the deposition. Besides, tip of temperature is covered with teflon tape to prevent any unwanted reaction.

3.2.2. Experimental Procedure

After the solutions are ready, experimental deposition setup is prepared by using non-soluble anode. For nickel sulfamate electroplating, current density is 40 A/dm^2. For the area, which is 4 cm^2, current should be 160 A constantly. One-hour coating process was carried out with cathodes which are prepared and insoluble anodes in homogenous solution with continuous mixing solutions at 50°C. Vortex formation was prevented by keeping the mechanical agitation rate constant during the experiment.

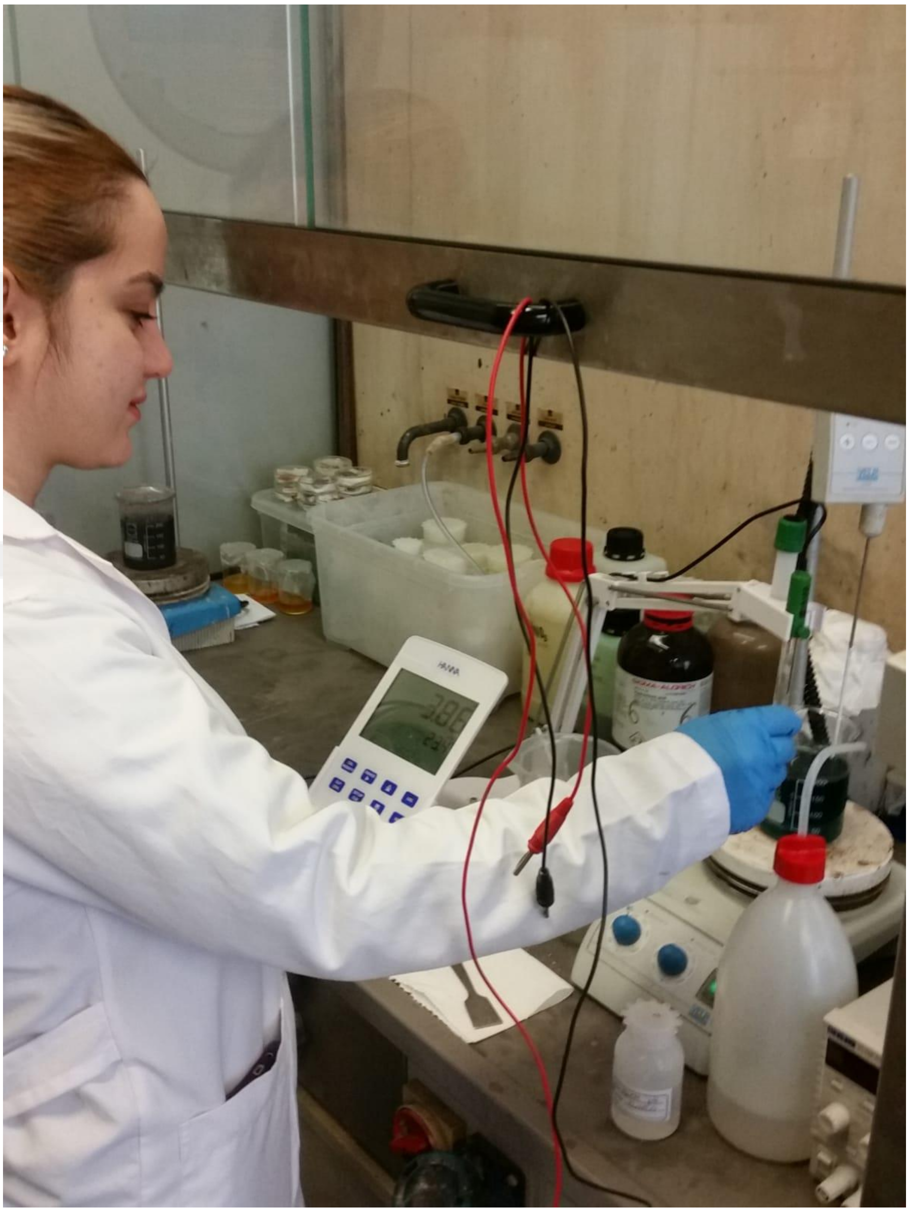

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Figure 8 - Experimental Setup

4. RESULTS AND DISCUSSIONS

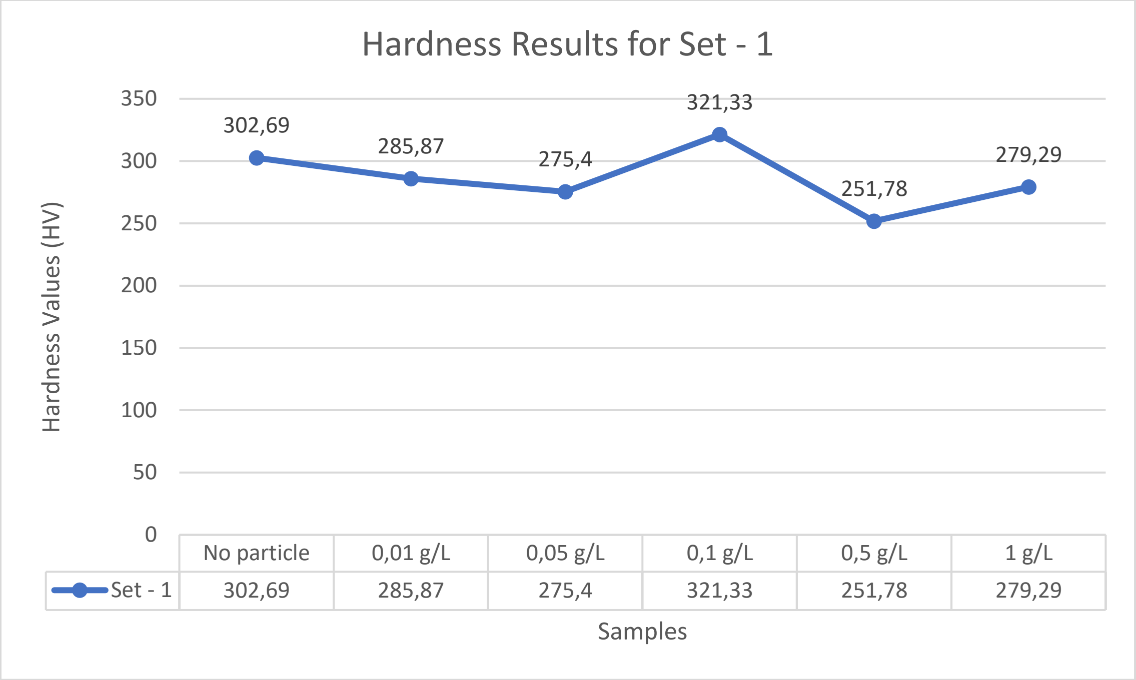
4.1. Hardness Results and Yield Strength

The hardness test results on six samples with different nanoparticle content are shown in the Table 18.

Table 18 - Hardness and Young Modulus Results for all samples

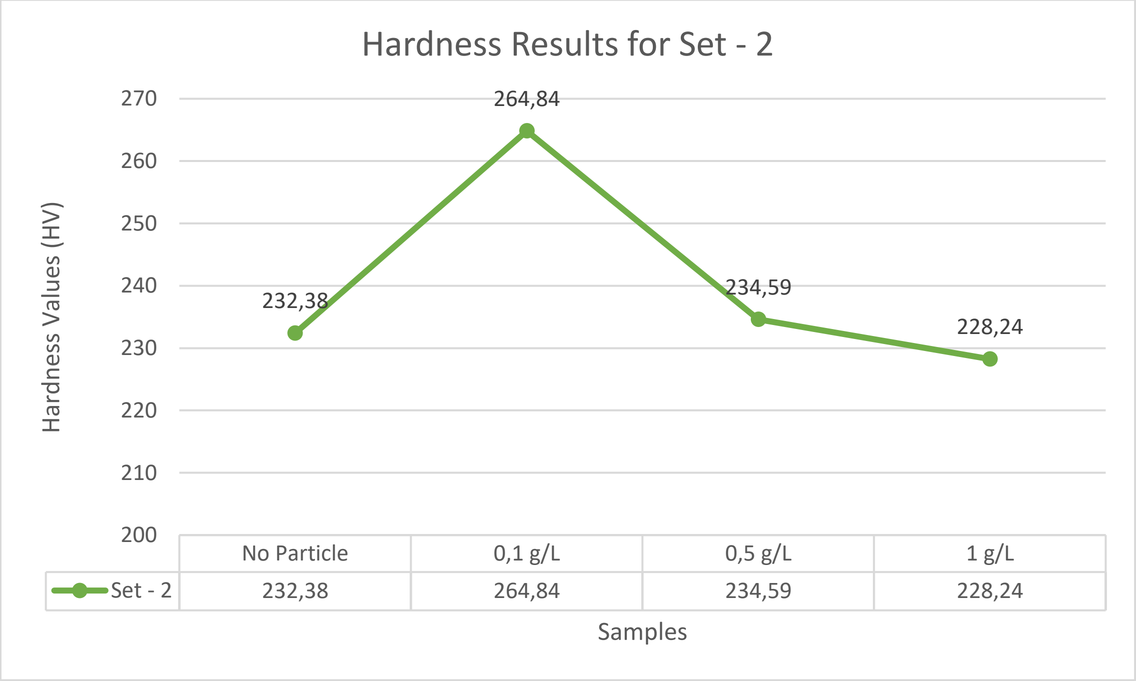
	Sample Labels	**Hardness Results (HV)**	**Young Modulus (GPa)**
Set - 1	S1	302.69	137.92
	S6	285.87	132.03
	S8	275.4	132.84
	S3	321.33	170.21
	S4	251.78	137.86
	S2	279.29	133.55
Set - 2	S7	232.38	99.14
	S5	264.84	146.38
	S10	234.59	109.02
	S9	228.24	126.16
Set - 3	S11	294.4	93.23
	S12	297.95	138.53
	S14	319.1	102.29
	S13	314.99	112.55

Silicon carbide nanoparticles affect the hardness of coating layer in two different ways. It increases the hardness of the coating by transferring its hardness to the material and changes the structure of the coating by affecting the formation of silicon carbide particles grains. This indirectly changes the structure of the coating and affects its hardness. The sample which contain 0.1 g/L silicon carbide nanoparticle is approximately 6.16% harder than the sample that without silicon carbide nanoparticle.



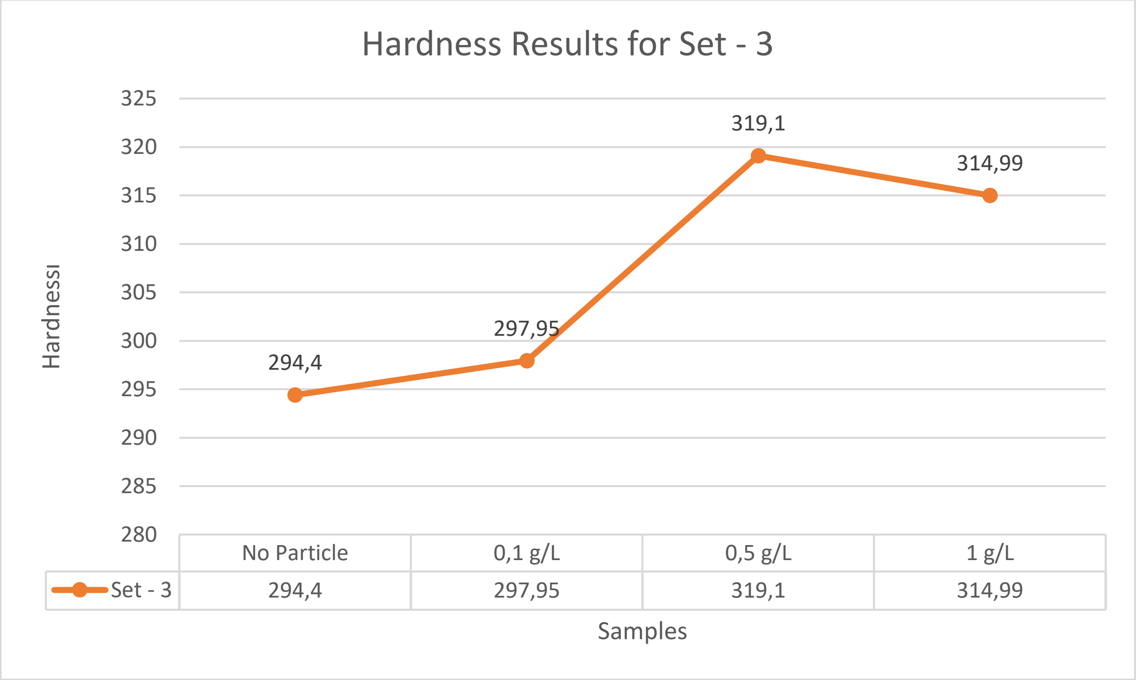
Graphic 1 - Hardness Results for Set – 1

Samples of Set – 2 which are made from the same solutions of Set – 1 with lower pH value and pH which is one of the important operational parameters of electroplating has effects on hardness of coating layer show that the sample which contain 0.1g/L silicon carbide nanoparticle is harder than no particle sample with a percentage of 13.97.



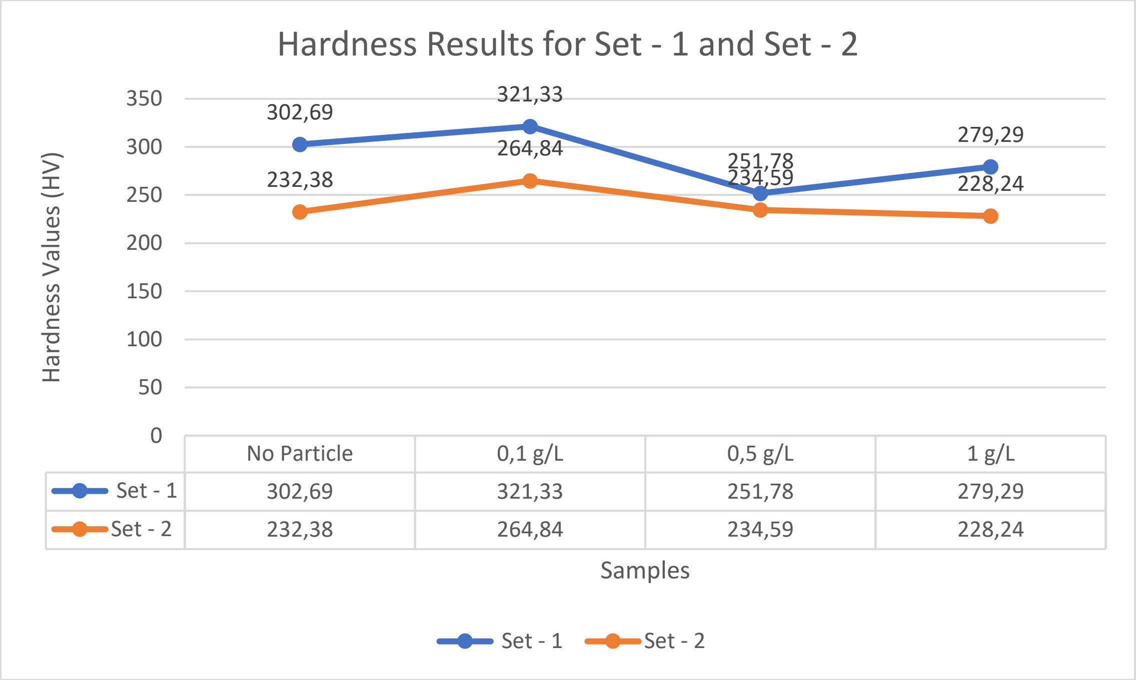
Graphic 2 - Hardness Results for Set – 2

Samples of Set - 3 made to repeat 4 samples produced in Set - 1 which are S1, S2, S3 and S4 were reproduced from the same solution of Set - 1 samples.



Graphic 3 - Hardness Results for Set – 3

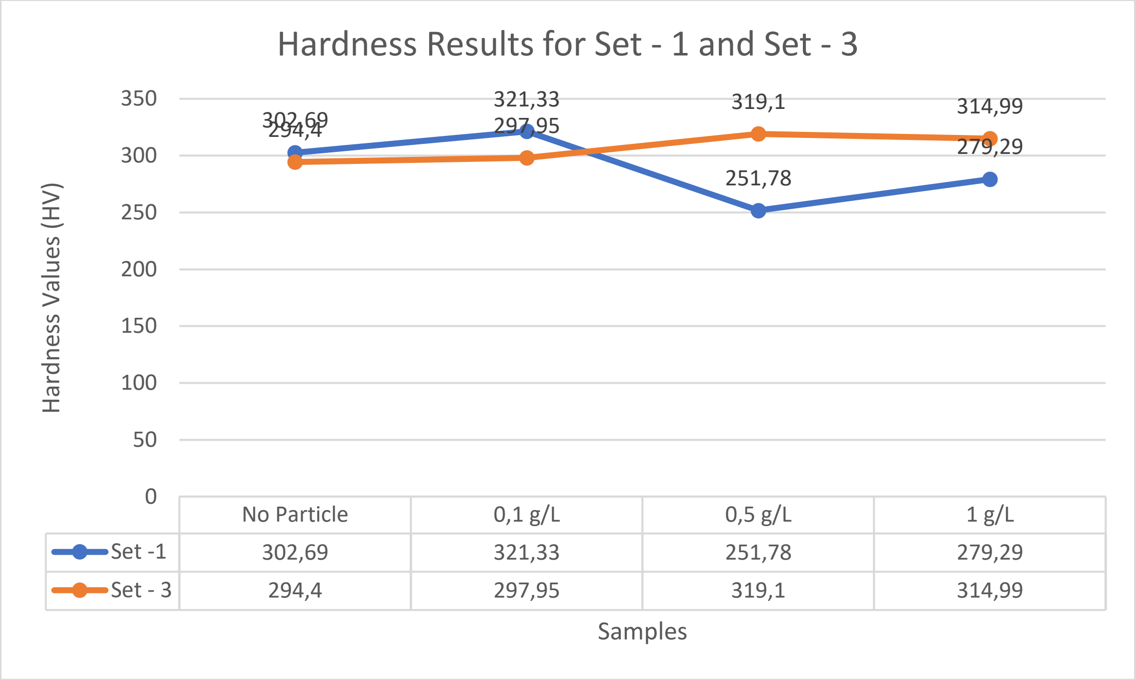
When the samples which have different amount of silicon carbide nanoparticle and the sample without contain nanoparticle were compared, silicon carbide nanoparticle is increased the hardness of coating even the solutions are used for several times.



	No Particle	0,1 g/L	0,5 g/L	1 g/L
Set - 1	302,69	321,33	251,78	279,29
Set - 2	232,38	264,84	234,59	228,24

Graphic 4 - Comparison of Hardness Results of Set - 1 and Set – 2

Besides, in order to observe the pH effect on hardness samples of Set – 2 show that, the hardness value of 0.1g/L silicon carbide nanoparticle samples were compared and the sample from Set – 2 has approximately 21.33% lower hardness value than Set – 1. Therefore, when pH is lower than optimum value for plating, hardness value of coating layer is decreases.

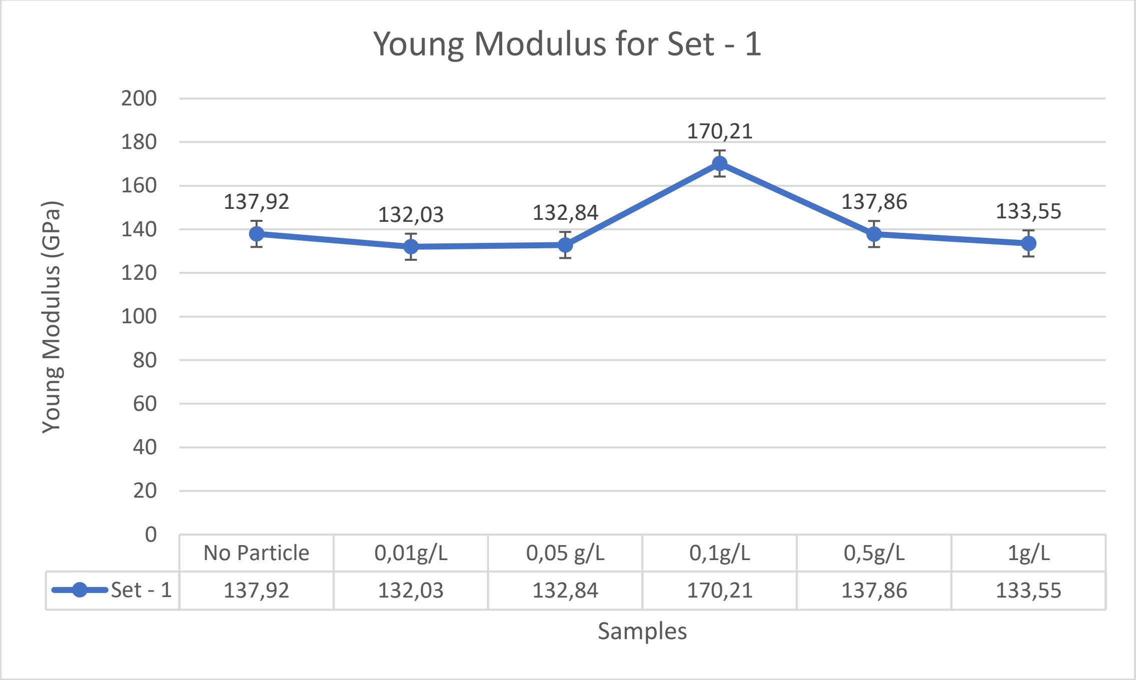


	No Particle	0,1 g/L	0,5 g/L	1 g/L
Set -1	302,69	321,33	251,78	279,29
Set - 3	294,4	297,95	319,1	314,99

Graphic 5 - Comparison of Hardness Results of Set - 1 and Set – 3

The sample which contain 0.1g/L silicon carbide nanoparticle from Set – 1 is harder with a percentage of 7.85 than the same sample from Set – 3. This results here show us the effects of decreasing amount of nanoparticle in the same solution on hardness.

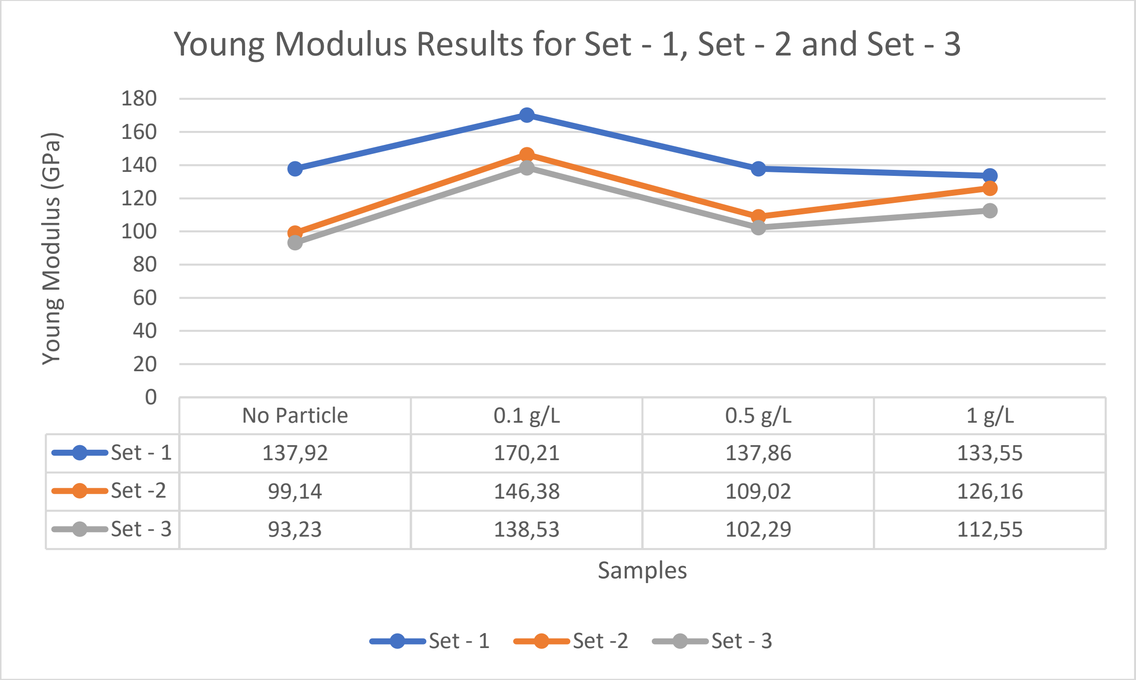
According to hardness results, young modulus of samples can be measured from the same test machine.



	No Particle	0,01g/L	0,05 g/L	0,1g/L	0,5g/L	1g/L
Set - 1	137,92	132,03	132,84	170,21	137,86	133,55

Graphic 6 - Young Modulus Results for Set - 1

While the sample which contain 0.1g/L silicon carbide nanoparticles has highest young modulus value which is 170.21 GPa, this situation continued when the experiment was repeated 2 times more.



	No Particle	0.1 g/L	0.5 g/L	1 g/L
Set - 1	137,92	170,21	137,86	133,55
Set -2	99,14	146,38	109,02	126,16
Set - 3	93,23	138,53	102,29	112,55

Graphic 7 - Comparison of Young Modulus Results for Set - 1, Set - 2 and Set - 3

When the young modulus results of all samples were compared, samples which have 0,1 g/L silicon carbide nanoparticles increased the young modulus with compared to samples without silicon carbide nanoparticles was observed. Young modulus of coating layers was increased with a percentage of 23.41 for Set – 1, percentage of 47.65 for Set – 2 and percentage of 48.59 for Set – 3 samples.

4.2. AFM

For the characterization test of AFM, 2 solutions which are one is the without silicon carbide nanoparticle and the other is with contain 0.1g/L silicon carbide were selected and 3 samples were produced on copper wafer with 1 and 3 seconds to observe the nucleation and thickness of deposit.

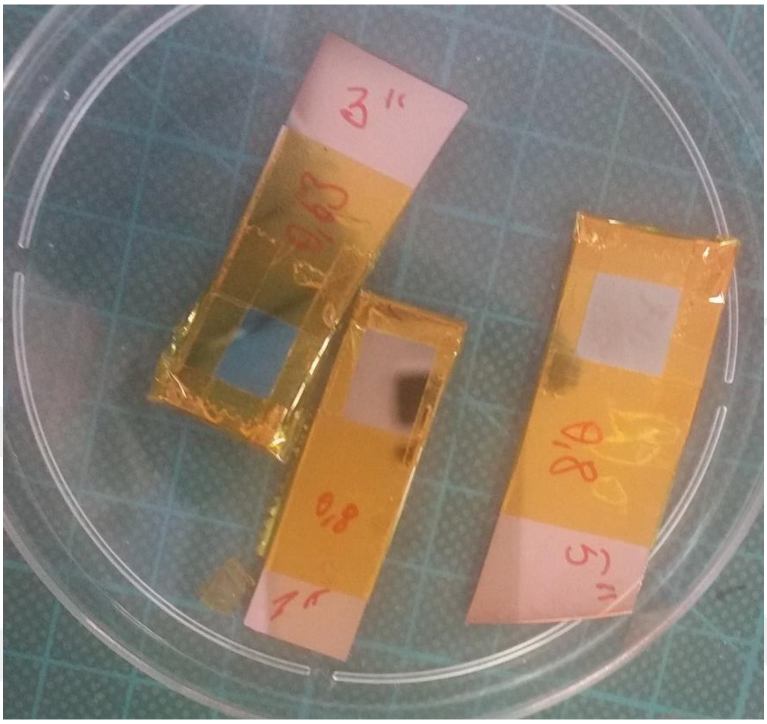
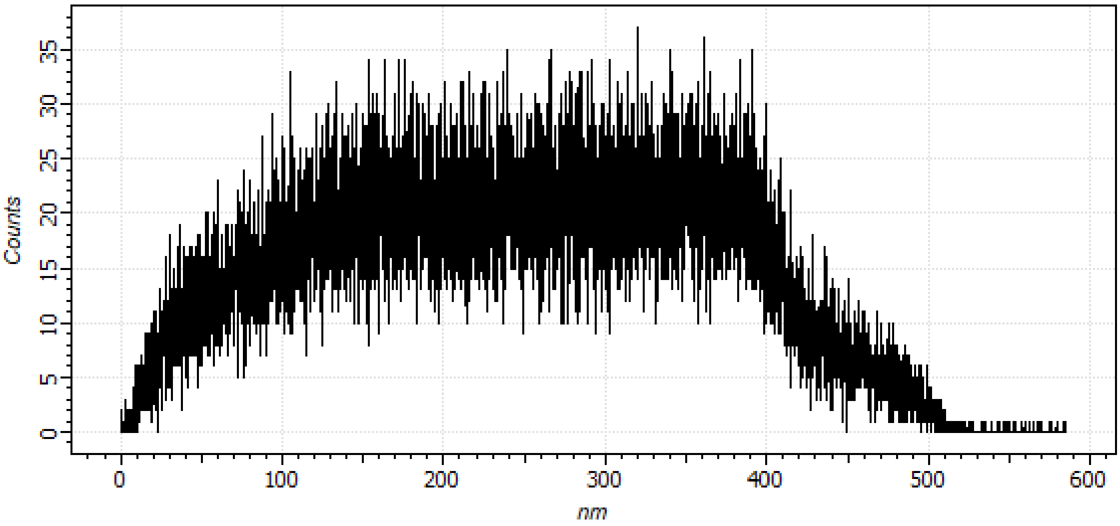
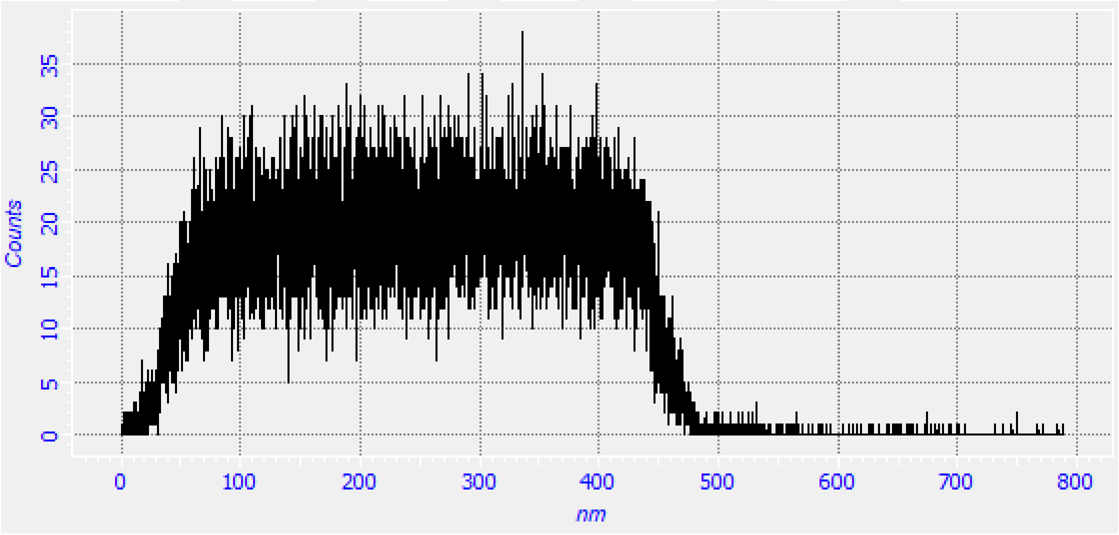


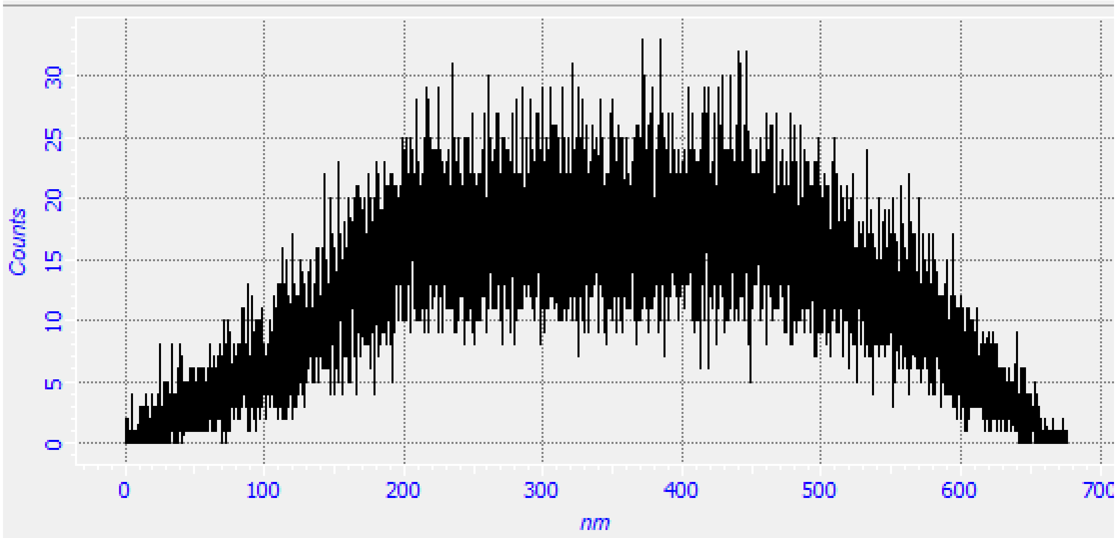
Figure 9 - Samples for AFM



Graphic 8 - Roughness Analysis of a Sample without nanoparticle for 1 second



Graphic 9 - Roughness Analysis of a Sample without nanoparticle for 3 seconds



Graphic 10 - Roughness Analysis of a Sample with silicon carbide nanoparticle (0.1g/L) for 1 second

The results obtained from the roughness analysis have been observed that silicon carbide nanoparticles help to obtain a more uniform coating across the surface of the material.

Table 19 - Roughness Analysis Data

Data of Roughness Analysis	**No particle for 1s**	**No particle for 3s**	**0.1g/L SiC for 1s**
Amount of Sampling	65536	65536	65536
Max	585.338 nm	788.923 nm	676.744 nm
Min	0 nm	0 nm	0 nm
Peak to peak, S_y	585.338 nm	788.923 nm	676.744 nm
Ten-point height, S_z	290.225 nm	386.923 nm	337.406 nm
Average	245.941 nm	250.596 nm	348.197 nm
Average Roughness, S_a	99.7371 nm	102.753 nm	121.401 nm
Second Moment	272.575	277.741	376.842 nm
Root Mean Square, S_q	117.516 nm	119.757 nm	144.113 nm
Surface skewness, S_{sk}	0.000466218	-0.000364294	-0.0487848
Coefficient of kurtosis, S_{ka}	-0.933014	-1.00301	-0.848309
Entropy	11.8212	11.7886	12.1297
Redundance	-0.286214	-0.225184	-0.29035

5. CONCLUSION

During this study, composite coating, and nickel metal matrix composite coating by using silicon carbide reinforcement are tried to understand. After searching and reading the previous researches, this study mainly focused on the effect of silicon carbide nanoparticles on wear behavior of coating layer which is produced by electroplating from nickel sulfamate bath with contain silicon carbide nanoparticles.

Samples were examined in hardness machine and atomic force microscopy. As a result of the hardness tests made on the produced samples, the silicon carbide nanoparticles used as a reinforced material increased the hardness of the coating layer and thus wear resistance of samples increased. It was observed that the hardness increased in the coatings with 0.1 g/L content although there was a change in the operational parameters. It has been observed that the wear resistance of the coatings applied one after the other with the same solution increases. The silicon carbide nanoparticles used as a result of the tests carried out in the atomic force microscope enabled the coating to have a more uniform thickness on the surface. Also, according to the AFM results, nucleation could not be observed due to the high rate of deposition.

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