

A Review of Electroless Plating On Different Substrates

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Abstract - In this review, Electroless Plating has been thoroughly introduced. It consists of the various plating accessories, the method involved in the plating procedure, applications, properties and the tests that can be performed for the material's characterization. In relation to the wear resistance, the conception of properties of coating is necessary in order to block the development of acute damages. Here, the implementation of Zirconium Dioxide (ZrO_2) layer on different substrates by using Electroless Plating is studied. It focuses on the protective coating of a substrate against crack propagation, oxidation and the corrosion. Unlike other coatings the Zirconium Dioxide (ZrO_2) also known as Zirconia is a material that is highly resistant to the thermal expansion.

Key Words: Electroless Plating

1. INTRODUCTION

Electroless Plating was first discovered by a French Chemist named Charles Adolphe Wurtz with Hypophosphite that caused an accidental chemical reduction of Nickel salts to Nickel metal in the year 1844 but was later patented by Francois Auguste Roux as a metal plating process in the year 1914. Although, even after receiving the patent Roux's invention was not much commercially successful. After the 2nd World War, in the year 1946 this plating technique was accidentally re-discovered by Abner Brenner and Grace E. Riddell of the National Bureau of Standards (NBS), United States of America. They developed a method for plating the internal tubular walls with an alloy of Nickel-Tungsten, procured from a Citrate Bath, utilizing an insoluble anode to bring the properties of Hypophosphite. Chemical reduction of Ni ions was occurred by virtue of this plating process. This modified discovery of Brenner and Riddell was more recognized than the early two discoveries made by Wurtz and Roux and got accepted worldwide. From that day on Electroless Plating made its way into an official existence. It got its recognition as a plating technique that runs without electricity and uses the action of chemicals to do the job of an anode, a cathode and cyanide salt solutions. Compared with other plating procedures, the Electroless Plating process had been slightly quiet younger being economically accessible. However, in the past 10 years the use of coating had grown to such volumes that the Electroless Plated parts are established underground, launched in the outer space and in a myriad of applications. Today, Electroless Plating has grown into a very consequential section of the finishing industry.

Various natural properties of Electroless Plating, such as resistance to wear, non-magnetic properties, deposit uniformity, lubricity, solderability, bondability, corrosion resistance and finally electrical resistivity make this coating a choice for many industrial and commercial uses. Electroless Plating enhances the surface hardness of the material, supplies a dense coating and in many cases provides the same surface finish that the material had before the plating operation. The process of coating is the most effective method to improve the physical capabilities of a metal, by letting the mechanical properties of the substrate to be maintained while giving them protection against friction by means of a protective coating. A controlled autocatalytic deposition of a continuous uniform layer over a catalytic surface by the chemical reaction of a reducing agent and a complex compound is commonly known as Electroless Plating. It is a general method in which some metallic Ions are reduced in a solution. Here, the film deposition is being carried out by the oxidation of a chemical compound, that is a source which supplies an internal current.

2. EXPERIMENTAL PROCEDURE

Electroless Plating contains three fundamental techniques. These are generally cleaning, coating and washing. In the plating procedure, first of all we have to wash the metal substrate by chemical cleansers which separate oil and other unwanted elements that are corrosive. The aqueous solution comprises of Nickel Sulphate as a metal salt, Sodium Hypophosphite as a reducing agent and finally Sodium Acetate as a buffer solution. Now, we dip the material into the standard solution and add antioxidant chemicals like Chromium, Cadmium or Arsenic that develops a specific layer of coating which makes the component highly corrosion and frictional resistant. Zirconium Dioxide (ZrO_2) is applied into the aqueous solution. Maintaining the PH scale of optimum range as 7, the temperature is adjusted to 93°C.

3. ACCESSORIES

3.1 Bath

Any laboratory appliance, made up from a container filled liquid which can either be an acid, alkali or any solution that incubates a specimen at a constant temperature over a longer time period is known as a Bath. Its major functions include providing electrolyte for the electrical conductance, stabilis



ing the temperature and containing providing electrolyte for the electrical conductance, stabilising the temperature and containing the reducing agent. A bath can be classified into two types. The Acidic Bath and the Alkaline Bath. The common chemicals used in Acidic Bath are Boric Acid (H₃BO₃), Potassium Chloride (KCL), Zinc Chloride(ZnCl₂) and Manganese Chloride(MnCl₂) while the chemicals used in Alkaline Bath are generally Potassium diphosphate (K₄P₂O₇), Zinc Sulphate (ZnSO₄ 7H₂O) and Manganese Sulphate (MnSO₄). Similarly, some common substrates used in Acidic Bath are Glass, Steel and Grey Cast Iron whereas the substrates used in Alkaline Bath are Stainless Steel, Platinum, Carbon Steel and Copper in general.

3.2 Magnetic Stirrer

A magnetic stirrer also known as a magnetic mixer is a common laboratory equipment, that generates a rotating magnetic field to make the stir bar immersed inside a liquid spin very fastly. This rotating field may be produced either by a rotating magnet or a batch of stationary electromagnets, placed below the vessel. It is used for such cases where stirring rods and motorized stirrers are not systematically applicable. It generally contains a magnetic bar that is kept inside the liquid. This bar delivers the action of stirring. The bar's revolving motion is handled by a set of electromagnets mounted beneath the liquid container. The usual geometrical design of commercial Magnetic Stirrers is generally bar shaped with the usage of octagonal cross section. The limitation of Magnetic Stirrers is that it can only be used for only small experiments of 4 liters or less. Sometimes, stir bars face difficulties in dealing with viscous liquids. However, for more efficient stirring, variation of shapes exists. Magnetic Stirrers are widely used for gear driven motorized stirrers as they have an absence of external parts that can be worn out, less noise and greater efficiency. The normal speed of a Magnetic Stirrer generally ranges between 0 - 3000 RPM.

4. PROPERTIES

4.1 Physical Properties

4.1.1 Density

The compactness degree of a material is called Density. Density, also known as Volumetric Mass Density or simply Specific Mass is the mass per unit volume of a substance. A Pure Nickel density is nearly about 8.9 g/cm³. Electroless Nickel does not have a constant density. With increasing Phosphorus content, the density decreases significantly.

4.1.2 Melting Point

The temperature at which a state changes from solid to liquid is defined as the liquefaction point or the melting point. Pure Nickel has a melting point of about 1455°C but its melting point is highly affected by the Phosphorus content in an Electroless Nickel. However, the high temperature properties of a Pure Nickel are quite absent in an Electroless Nickel.

4.1.3 Deposit Uniformity

A great important characteristic for all fields of applications of Electroless Nickel is the production of deposits with an extremely high degree of thickness uniformity. It is certainly crucial while coating complex sections with critical geometry, such as ball valves or threaded portions. This huge advantage over Electrodeposited Nickel is due to the fact that no currents are involved.

4.1.3 Structure

Electroless Nickel's structure is certainly responsible for some of its distinctive properties. It differs from the ordinary Electrodeposited Nickel structure and can normally be described as having the ultra-fine crystallites or an Amorphous Structure. As the well-defined crystal structure is not present, the possibility of an intergranular corrosion is totally terminated. This can later be a problem for the crystalline deposits. For Ni-P deposits, the heat treatment can create significant changes in the crystallization process, can cause cracks in medium and high deposits of Phosphorous.

4.2 Electrical Properties

The fundamental quality that usually quantifies how strongly a material resists electrical current is commonly referred to as an Electrical Resistance. The resistivity of a Pure Nickel is generally about 7.8×10^{-6} ohm-cm, but that of an Electroless Nickel can be ten times higher. This mainly results due to the disruption of some of the regular lattice structures of Pure Nickel by the code position of Phosphorus. Eventually, the consequent resistivity of Electroless Nickel increases with the increasing content of Phosphorous.

4.3 Magnetic Properties

The typical magnetic properties of Electroless Nickel are extremely essential as they have been recognised as responsible for one of the largest applications of high Phosphorus deposits, usually like an under layer for some magnetic coatings in the production of memory discs. The response of Electroless Nickel largely varies with the Phosphorus content. The ferromagnetism associated with the Pure Nickel falls dramatically with the increase in the content of Phosphorus. 11% Phosphorus deposits are described as non-magnetic.



4.4 Thermal Properties

The gradual dimensional modification of a specific material is utterly described by the coefficient of thermal expansion. It often specifies the fractional change in the object size, per degree change in the temperature along with a constant pressure. Coefficient of thermal expansion of an Electroless Nickel generally ranges from 22.3 μ m/m/°C at 3% Phosphorus to 11.1 at 11 % Phosphorus. In comparison, the usual value of an Electrodeposited Nickel normally varies from 14 to 17 μ m/m/°C.

4.5 Mechanical Properties

4.5.1 Hardness

The hardness of Electroless Nickel is a very important feature in a number of successful fields of applications. It strongly depends upon the amount of Phosphorus. Knoop Microhardness values varies from 500 to 720 HK. The hardness of Electrodeposited Nickel remarkably diminishes by heat treatment process due to grain growth and recrystallization.

4.5.2 Wear Resistance

Electroless Nickel have comparatively better wear resistance because of their natural lubricity and high hardness. Wear resistance can be developed by good heat treatment and code position of any particulate matter such as Polytetrafluoroethylene, Fluorinated Carbon, Alumina, Diamond and Silicon Carbide. Electroless Nickel can be used to solve different wear related issues.

4.5.3 Ductility

The deposition of Electroless Nickel undergoes lower ductility and Phosphorus quantity within a standard operating range, that has a very little effect on it. The coatings are generally hard and brittle. The elongation typically ranges between 1 to 2.5%. In order to increase hardness and reduce ductility, normal heat-treatment procedures are required.

4.5.4 Internal Stress

Deposits of Phosphorus are compressively stressed when they are plated from a fresh bath but usually the stress becomes progressively tensile as the bath is kept for a longer period of time. Low Phosphorus deposits are in absolute compressive stress whether aged or new. Those with medium Phosphorus contents are generally in tensile stress.

4.5.5 Corrosion Resistance

Electroless Nickel's corrosion resistance property is one of the major causes for its widespread utility as a successful protective coating nowadays. Other facts that must be taken into consideration in order to enhance the corrosion protection includes the nature of surface condition of the metal substrate, the deposit thickness and the heat treatment procedures.

4.5.6 Solderability

The ability of a material to be wetted by a molten solder is commonly known as solderability. In general terms, it is the measure of the ease with which a soldered joint can be made to that material. Depending upon the type of solder alloy used, solderability varies. In solderability, a wetting balance records and measures the wetting force as the time function. Metals are easy to solder but sometimes they have brittle joints. For soldering they require large amount of heat. However, the issue of oxidation can be overcome by using a flux.

5. APPLICATIONS

Chemical Process, Industry Chloralkali Industry, Steel Compressors, Control Valves, Saturated Brines, Transporting Brines, Cooling Towers, Cooling Tower Water Pumps, Nuts and Bolts, Rail Cars, Safety Vents, Unloading Connections, Air Connection Valves, Outer Valve Assemblies, Monochlorotoluene Columns, Grinding Screens, Pelletizing Equipments, Sea Water Cooling, Hot Taps, Engine Valves, Flow Metres, Food Industry, Packaging Equipments, Moulds and Rolls, Meat Processing Industry, Bakery Industry, Oil and Gas Industry, Flow Control Devices, Ball Valves, Choke Valves, Fire Flow Valves, Safety Valves, Khuff Gas Wells, Blowout Preventers, Tubular Components, Pumping Systems, Mud Pumps, Down Hole Drill Motor Rotors, Drill Bits, Automotive Industry, Alternate Fuels, Carburettors, Fuel Injection Systems, Aluminium Fuel Filters, Differential Pinion Shafts, Viscous Coupling Plates, Cast Iron Slip Yokes, Carburettor and Clutch Parts, Bearings and Gears, Aerospace Industry, Compressor Sections, Compressor Stator Assemblies, Compressor Spacers, Compressor Cases, Variable Vanes, Bearing Housings, Bearing Supports, Fuel Lines, Airframe Assemblies, Landing Gears, Navigational Systems, Metallic Optics, Actuators, Missile Guidance Systems, Space Shuttles, Electronics Industry, Memory Discs, Semiconductors, Printed Circuit Boards, Microwave Components, Transistor Chips and EMI Shieldings.

6. RESEARCH METHODOLOGY

6.1 SEM Analysis

SEM also known as Scanning Electron Microscope is a type of an instrument that scans the surfaces with a focused electron beam of relatively low energy in order to produce images of a sample. The particular specimen experiences an electronatom interaction which create various signals that contain information about composition and surface topography of the sample. There is an electron column present in the microscope that generate and arrange electrons through magnetic and electric fields. Under the influence of vacuum, the entire operation is exercised so as to avoid the collision between gas molecules and electrons. Finally, an image is built in the monitor. Basically, SEM runs on the principle of kinetic energy to generate signals on the interaction of Backscattered Electrons and Secondary Electrons. Here, the electrons generated by



radiation as ionization products like photons or ions are called Secondary Electrons while high energy electrons originating from a beam of electrons that are reflected back from the specimen by specimen atoms with elastic scattering interactions is simply known as Backscattered Electrons. The images taken by SEM has certain features like compositional differences, physical differences like shape or inclination, surface topography or surface texture, electric and magnetic fields. Various receptors are found in Scanning Electron Microscope. Chemoreceptors, that are stimulated by changes in the chemical concentration of substances. Pain receptors, that are stimulated by tissue damage. Thermoreceptors, that are stimulated by changes in the temperature. Mechanoreceptors, that are stimulated by changes in the pressure or movement. Photo receptors that are stimulated by light energy.



Fig - 1: Scanning Electron Microscope. [29]



Fig - 2: Secondary Electrons & Backscattered Electrons.
[30]



Fig - 3: SEM Image of Copper after Electroless Plating. [31]



Fig - 4: SEM Image of Plain Carbon Steel after Electroless Plating. [32]



Fig - 5: SEM Image of Aluminium after Electroless Plating.
[33]



6.2 EDX Analysis

EDX, sometimes called as EDS or simply Energy Dispersive X-Ray is a micro analytical technique which is used for the elemental analysis or chemical characterization of an area of a sample. EDX is basically used to analyse the elemental concentration that is present in the sample. It generally stimulates the sample by high energy photons or electrons and detects the material of outgoing photons. In EDX we are getting the atomic presence of a particular atom in the specimen. The output of EDX Analysis is called the EDX spectrum plot. The plot identifies the elements corresponding to each of its peak that corresponds to various energy levels. Here in the EDX spectrum plot the peak depends upon the amount of excitation of the atomic energy level. Higher the peak of the spectrum, it means that the element is more concentrated in the specimen. From EDX Analysis we usually obtain a curve that is the Intensity (Watt per metre square) v/s Wavelength (metre) curve. The peak of the curve we obtain is commonly known as an X-Ray Continuum. Determination of chemical elements present in a material can easily be achieved utilizing the key features of EDX. The micro analytical technique can estimate the relative abundance of those elements. The test also helps to achieve the dimensional data from multi-layer coating thickness of metallic coatings. However, the accuracy of this quantitative analysis of sample composition is strongly dependent upon a number of factors. The accuracy of the composition measurement is totally affected by the characteristics of the metal sample. When any atom in a material is sufficiently excited by an incoming beam generation of X-Rays occur.



Fig - 6: Energy Dispersive X-Ray Machine. [37]



Fig - 7: EDX Spectrum Plot. [38]

6.3 Nano Indentation Test

In Nano Indentation Test an indenter is forced into the specimen in a continuous or a stepwise mode. The load and displacement of the indenter are measured and recorded during loading and unloading. The depth varies between several micrometres and nanometres. In this test the calibrated indenter approaches to the sample surface. After the sample contact, the force is slowly increased with linearity and the tip finally indents into the surface of the specimen. The point of contact is determined by the force-displacement data. PH curves obtained calculate nano mechanical properties of thin coating like hardness, fracture toughness, plastic energy and elastic energy. Here, elastic energy is the energy subjected to elastic deformation while plastic energy is the energy subjected to plastic deformation. Nano Indentation Test is needed for certain purposes. As we are using nano particles or materials in coating, we cannot go for any ordinary hardness tester because we need to have a micro hardness. For micro hardness we need to get the result on different loading and unloading. This is what, that is mainly done in a test like Nano Indentation Test. The load-displacement curve is recorded during a Nano Indentation Test. The thermal drift is eventually measured by means of some obtained data at maximum load or final unloading point. Events like phase changes and cracking are identified as features on the curve of load displacement. It is necessary to display the load-displacement data with a load on the vertical axis. When thermal equilibrium is established, the testing proceeds. By mounting the device on an anti-vibration table the mechanical vibration is minimised.





Fig - 8: Nano Indentation Tester. [41]



Fig - 9: Load-Displacement Curve of a Nano Indentation Test. [42]

6.4 Nano Scratch Test

Nano Scratch Test is a flexible tool for the analysis of both bulk materials and thin films. Nano Scratch Testing readily investigates fracture and deformation. Here, in Nano Scratch Test a normal load is applied in a controlled fashion while measuring the force required and by moving the tip laterally across the sample. Henceforth, selecting appropriate lateral displacement pattern and normal loading profile different types of tests can be done. To perform those tests, different types of accessories and apparatus like T1980 Triboindenter, T1 Premier, T1 77 Select, P1 88 SEM Picoindenter, P1 95 TEM Picoindenter and TS 75 Triboscope are required. For thin films, the scratching strength is normally measured by a particular diamond tip which linearly scratches the thin film under a vertically downward load applied from the above in a Nano Scratch Test. The frictional coefficients of thin films, tip loads in the lateral and vertical directions, tip displacements and etc. can be obtained from this test. Nano scratch test can be used for the evaluation of various adhesive properties. Scratch properties of thin films can be measured easily

by a Nano Scratch Test in comparison with other conventional scratch methods. Nano Scratch Test is suitable for featuring the practical adhesion failure and crack resistance of coatings and thin films with a thickness less than 1000nm. The test can be used for analysing inorganic and organic coatings as well as hard and soft coatings. The exceptional design of the measurement head contains double sensors associated with a piezoelectric actuator. One sensor for the depth measurement and the other one for measuring force. However, ultrathin layers of less than 10 nm are not suitable for measurement.



Fig - 10: Nano Scratch Tester. [46]



Fig - 11: Load-Displacement Curve of a Nano Scratch Test. [47]

7. COMPARISON OF ELECTROLESS Ni-P, Ni-B AND Ni-BP COATINGS

7.1 In terms of Corrosion Resistance

Compared to Sodium Hypophosphite which is used as a reducing agent for depositing Ni-P coatings, Sodium Boro-Hydride or Amino Boranes are used instead as reducing agents to achieve Ni-B coatings. Ni-P coatings exhibit good



corrosion protection applications because of its higher corrosion resistance, while Ni-B coatings show greater wear resistance, better adhesion and a comparatively lesser corrosion resistance. Electroless Ni-BP coatings have slightly better corrosion resistance and wear resistance than that of the Electroless Ni-P coatings.

7.2 In terms of Voltammogram Measurement

There are two anodic peaks for Ni-B solution. With the addition of DMAB in the Nickel containing Ni-P solution, a new anodic peak appears. In Ni-BP solution there are two anodic peaks and one cathodic peak similar to the Ni-P bath. The initial reduction potential of Ni-BP bath is shifted towards the positive direction. However, the initial reduction potential of Ni-BP solution is more negative than that of the Ni-P and Ni-B solutions.

7.3 In terms of Morphology

All deposits are invariable and free of cracks. Ni-P deposits possess spherical nodular structures with good uniformity and denser coverage. The cross-sectional appearance of the Ni-P is quite amorphous in all nanocrystalline structures. The morphology of Ni-B coatings relates to a cauliflower morphology or colunar morphology. Ni-P morphology is more dominant than Ni-B morphology. However, the nodule size of Ni-BP deposits is generally larger than that of Ni-P deposits, since the growth rate and nucleation of nodules could be suppressed and nodule coalescence is occurred with the introduction of even small amount of Boron. A nanocrystalline structure is obtained in the Ni-BP ternary system. In spite of low Phosphorous and Boron content in Ni-BP deposits the generation of nanocrystalline structure might probably be due to a the Phosphorous and Boron dissolution at the grain boundaries. Ni-P and Ni-B deposits will become nanocrystalline when the Phosphorus and Boron contents will be above 7 wt.% and 1.5 wt.%, respectively.

7.4 In terms of Hardness

The hardness of Electroless Ni-P and Ni-B coatings is dependent on the Phosphorous and Boron content. Although the hardness of Ni-P coating decreases with the increase in Phosphorous content, the hardness of Ni-B coating increases with the increase in Boron content. Ni-B coatings have higher hardness values than that of the Ni-P coatings. After heat treatment process the hardness values of Ni-P, Ni-B and Ni-BP coatings reach to 7.08, 10.02 and 9.92 GPa respectively. After heat treatment, the average grain size of Ni-P, Ni-B and Ni-BP coatings are obtained as 15 nm, 10 nm and 11 nm respectively. Ni-BP coatings show higher hardness value than that of the Ni-P and lower hardness value than that of the Ni-B coatings. The reason for the lowest hardness value of Ni-P than that of the Ni-B and Ni-BP is probably related to higher grain size and Ni-BP precipitations have softer precipitation compared to the Ni-BP precipitations. Also, Ni-BP coatings exhibit similar hardness behavior with Ni-P. The difference between Ni-BP and Ni-P is that Boron produces a solid solution in Ni domain which causes an increase in the hardness of the coating Ni-BP that shows lower hardness value than that of the Ni-B after heat treatment process.

7.5 In terms of Wear Resistance

Ni-BP coatings are expected to show better wear resistance than Ni-P and Ni-B coatings. It is evident that Ni-BP coating has consistently shown better wear resistance throughout the range of investigated parameters. Furthermore, the wear rate of the Electroless Ni-P, Ni-B and Ni-BP depositions decrease with the increase in the sliding velocity under all normal load conditions. Moreover, the wear rate decreases from 1.6×10^{-10} ⁵ to 2.18×10^{-7} mm³ /Nm when the sliding velocity varies from 100 to 400 m/s at a normal load of 1 N. At the velocity of 100 mm/s and a normal load of 1 N, Ni-BP deposition displays 36.5% enhancement in the wear resistance as compared to the Ni-P coatings whereas the Ni-B demonstrates only 10.7% improvement. At the velocity of 100 mm/s and a normal load of 2N, the Ni-BP deposition shows 58% improvement in the wear rate as compared to the Ni-P which only has 12.2% improvement in the wear rate. As a result, the Ni-BP coating displays the lower wear rate among all applied load and sliding velocities which might be due to the high H/E ratios when compared to the Ni-B and Ni-P coatings. It is clearly observable that the Ni-BP depositions possess the highest H/E ratio than the other coatings. However, the wear rate of Ni-P deposition is higher than Electroless Ni-BP deposition.

8. OBJECTIVES

The review of Electroless Plating experiment for various substrates using Nickel Phosphorus (Ni-P) coating with Zirconium dioxide (ZrO_2) as a Nano Particle is conducted. After plating, the drastic improvement of mechanical and corrosive properties can be accomplished. Material characterizations like SEM Analysis, EDX Analysis, Nano Indentation Test and Nano Scratch Test can be performed.

9. FUTURE SCOPES

TEM Analysis, XRD Analysis, OCP Test, Wear Test and Cavitation Erosion Test.

10. CONCLUSION

Due to its superior corrosion resistance and performance enhancing capabilities the hurdle of corrosion will largely be annihilated by the development of Electroless Plating technique. Due to its reliability decreased cost, Electroless Plating can be considered for various commercial applications. There will be a prospective for the Electroless Plating process to



play a very important role in the fields of electronic industry, micro and Nano devices in the nearest future.

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Volume: 05 Issue: 08 | Aug - 2021

ISSN: 2582-3930

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