

Nickel Nano Particle Composite by Electrodeposition Method

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Abstract: A turbine blades are the individual component which makes up the turbine section of a gas turbine or stream engine. The turbine blades are responsible for extracting energy from the high temperature, high pressure gas produced by the combustor the turbine is often the limiting component of gas turbine to survive in this difficult environment.

Nickel coatings are commonly applied to steel, Nickel and other metal to provide protection against corrosion and abrasion, however most nickel is consumed in decorative finishes, our main aim is to change the characteristics of turbine blades which can withstand high temperature and availability of the material. At present they are using Ni and Rh composite material, we are replacing nickel with mild steel which is easily available in nature and we are giving Ni coating on it.

Keywords: Electrodeposition.

1. Introduction

The Electro deposition method of corrosion resistance of metals, alloys, and semiconductors has formed wide use in the fabrication of Microsystems and micro electromechanically systems (MEMS). A large number of metals found in Microsystems can be electroplated from aqueous electrolytes (Ni, Cu, Au, Pt, Fe, Pb, to name a few), except where a few metals cannot, notably Al, Ti, and pure W. Nickel and its high strength alloys provide some of the more useful materials available to Microsystems fabrications by Electrodeposition, owing to their greater uses in forming mechanical and magnetic elements such as precision gears, latches, motors, and flexure spring arms. Electroplating is a versatile technique spanning a wide range of coatings and thin films less than a micron to thick electroformed mechanical elements with millimeters tall. Varieties of materials are deposited near ambient conditions without the need for any of the expensive vacuum equipment. In this method deposition rates can be too faster than vapor deposition methods and film thickness can be as high as a millimeter or two. The necessary elements of the process are the cathode, or the work piece to be plated, an anode or counter electrode, and electrolyte that occupy the metal ion in a reducible form. While current is supplied to the work piece, which form MEMS is often a flat substrate metalized by vapor deposition. Through this thin metal coating forms a conductive seed layer to which electrical touch is created. This seed layer wrap micro patterned films of insulator, as in the copper

Damascene process parts, illustrate by the LIGA (German acronym associated with the process of litho graphically defining moulds for electroforming) and X-ray lithographic methods. The use of LIGA process overcomes many disadvantages of conventional fabrications methods and pattern transfer technique like chemical etching, sputter etching, reactive ion etching.

A. Electrodeposition

In the process of electrolysis, the cell circuit consists of an anode (positive electrode), the cathode (negative electrode), an electrolytic bath, a current source and an ampere/ voltmeter. Reduction and Oxidation take place at the cathode and anode respectively because of the metal ions and electrons that can cross electrode-electrolyte interface. Where cathode is conducting substrate on which Electrodeposition required to be done; the anode can be either soluble or stagnant. The overall reaction occurring during electrolysis can be represented as Eq. 1 at the cathode, Eq. 2 at the soluble anode and Eq. 3 at insoluble anode:

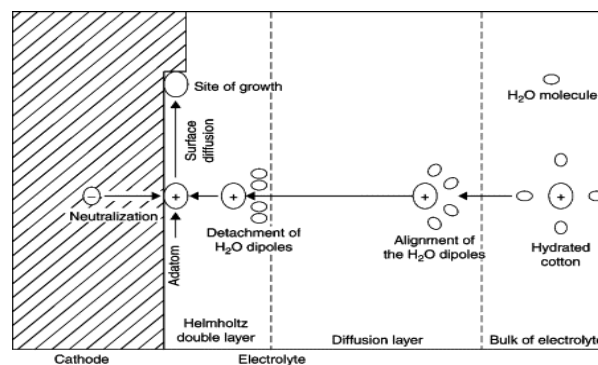
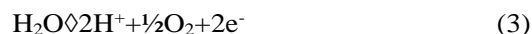


Fig. 1. Schematic representation of cathodic deposition

In the simple salt solutions, metal ions are present in mass solution as hydrated ions; the metal ions when hydrated ions as denoted as $M(H_2O)_x^{z+}$, where x is the number of water molecules in the primary hydration case. The reactions taking

place in the discharge process of ions under the effect of an electric field are the moving of the hydrated ions in the direction of the cathode surface, the arrangement of water molecules in the diffusion layer, the elimination of water molecules in the Helmholtz layer, released followed by decrease of the ions at the cathode surface as 'adatoms', surface distribution and the abstraction of adatoms into the crystal lattice at the growth point. A diagrammatic representation of these steps is given in Fig. 1.

B. Corrosion and protection of metals

Since, the last few decades, an extensive effort has been made to understand corrosion phenomena with their mechanisms, and to make clear the causes that dramatically impact the service lifetime of metal materials. The behavior of the materials in violent environments is evaluative for a substantial society. The washouts of the material in service have a great impact on the economy, the environment, health, and society. In the regard, corrosion-based financial losses due to maintenance, repair, and the exchange of existing structures and infrastructure account for upto 4% of gross domestic product (GDP) in well developed countries.

One of the major drawbacks in corrosion engineering is estimating service lifetime. Corrosion judgment has become very difficult, as there is no correlation with service lifetime and experimental lab outcomes, usually as a result of variation between accelerated testing and real corrosion process. It is most important to forecast the impact of corrosion-based losses on society and global economy, since ancient structures and infrastructure are becoming outdated, and critical decisions now need to be taken to replace them.

While, on the other hand, environmental agreements aim to reduce greenhouse effects. Consequently, low emission policies, in force, setup regulations for the future generation of materials and technologies. Advanced technologies and budding materials will permit us to get via the next century. Great supports are currently made in progress for the development of corrosion-resistant metal materials for each sector, such as energy, transport, construction, health, domestic appliances and electrical equipments.

And special issue on the corrosion and protection of metals is focus and observation on current trends in corrosion science, engineering and technology, scaling from fundamental to applied research, thus developing subjects related to corrosion mechanism and modeling, protection and inhabitation processes and mitigation strategies.

C. Mild steel and its applications

Low carbon steels are used as the preferred construction material across industries and are considered the more economical option than the costly corrosion-resistant alloys. Carbon steels typically contain less than 1.5% carbon content along with the minute presence of Mn, Si, P and S. Based on the percentage of carbon, the classification is further divided into three forms, namely low carbon steels (<0.25% C),

medium carbon steels (0.25-0.70% C) and high carbon steels (0.70-1.05% C). Variation of the percentage of carbon content allows to attain different mechanical properties such as strength, ductility, hardness, corrosive properties etc.

Carbon steels have wide range of applications, such as structural components, industrial pipes, and kitchen appliances. With regards to applications in the oil and gas industry, the two major forms of corrosion are carbon dioxide (CO₂) corrosion, which is also known as sweet corrosion, and hydrogen sulphide (H₂S) corrosion which is commonly known as sour corrosion.

D. Corrosive properties of mildsteel

Mild steel has been used widely in the oil, gas and chemical industries because of its excellent mechanical properties. Using mild steel is also one of the effective approaches to maximize profit and minimize cost as compared to high cost corrosion resistant alloys. Anyhow, this alloy still suffers from corrosion when it comes in exposure with corrosive environments, generally those carrying chloride ions. In many industrial applications allied to oil and gas processing such as pipeline cleaning, pipeline/acid descaling and oil well acidizing, the use of mineral acids (usually Hydrochloric acids) is still a strong method for improving productivity. Productive ways to prevent corrosion of metals modified by some industries include material selection, and cathodic protection amongst others. Although, these methods are highly expensive, which is a difficult in practical application as per as acidic media is concerned, the use of organic inhibitors is one of the experimental methods to action the hazard of corrosion due to its advantage of economy and high efficiency.

2. Methodology

Methodology deals with the systematic representation of the methods used for research and analysis. This project helps in development of metals towards resistance of corrosion of metals towards acidic environments, and hardness of metal for deformation for minimal change in shape. Mild steel is an alloy which has great revolution in structural and industrial applications due to low carbon content with low tensile, easier availability and cheaper cost material. These properties of mild steel are made to use them in constructing building and bridges etc. Poor corrosion resistance of mild steel is serious concern in many industrial applications. Since last few decades, efforts have been made to understand corrosion phenomena and their mechanism, and to elucidate the causes that dramatically influence the service lifetime of metal materials. Application of corrosion resistance metal coating is possible solution to combat steel corrosion. The main purpose of the project is to develop better relation between metals so as to deal with corrosion properties of the different metals. Through this we can build better metals that can help in reduction of economic worries by increasing the life time of metals. In order to develop these types of metals different properties of metals is to be studied. Also, different mechanisms that can help reduce

corrosion of these metals have been revised. With the conclusion of those mechanism we conclude Electrodeposition is better method that reduce corrosion with less economic invest and faster method.

3. Materials

A. Steel – Carbon Steel

It is an alloy of iron and carbon, with carbon content up to maximum of 1.5 – 2.0%. The carbon occurs in the form of iron carbide, because of its ability to increase the hardness and strength of the steel. Other certain elements like silicon; phosphorous, sulphur and manganese are also present in greater or lesser amount to impart certain amount of desirable properties to it. Most of the steels produced recently are the plain carbon steel or carbon steel. Carbon steel is defined as steel which has its properties mainly due to its carbon content and does not contain more than 0.5% of silicon and 1.5% of manganese. The plain steels are usually classified in their carbon content, the commonest of the range being mild steel carbon content.

Mild steel and cast iron are selected for this work. Mild steel is a carbon steel typically with a maximum of 0.25% carbon, 0.4% - 0.7% manganese, 0.1% - 0.5% silicone and some traces of other elements such as phosphorous. It may also contain lead (free cutting mild steel) or sulphur (again free cutting steel called re-sulphurised mild steel). Mild steel is a general term for a range of low carbon (a maximum of about 0.3%) steels that have good strength and can be bent, worked or can be welded into an endless variety of shapes for uses from vehicles to building materials even some of your pots and pans.

B. Nickel anode

Nickel is a chemical element with the symbol Ni and atomic number 28. It is a silvery-white lustrous metal with a slight golden tinge. Nickel belongs to the transition metals and is hard and ductile

C. Clamps and leads

Clamps and leads are electromechanical devices used to join electrical conductors and complete an electric circuit. Most of these are having a gender - i.e. the male component, called a plug, connects to the female component, or socket. The connection either be removable, require a tool for assembly and removal, or severe as a permanent electrical joint between two pints. An adapter can be used to connect dissimilar connectors. These connectors generally consist of two classes of materials: conductors and insulators. Properties important to conductor materials are conductivity, mechanical strength, formability, and resilience.

4. Procedure

A. Electrodeposition of Zinc

The Electrodeposition of Nickel on a mild steel plate was conducted in a three- electrode system. The working electrode

of Mild Steel of (1 cm²) was used as the substrate. Plates of mild steel with a nominal composition of 99.732% wt Fe, 0.17% wt C, 0.045% wt P, 0.045% wt

Sand, 0.008% wt N were used as the substrate as cathode. A Pt counter electrode placed in cell. Meanwhile, a high priority Ni plate (99.98%) with the dimension of (2×4 cm²) was used as the anode. Both the surface of both plates were polished using a common metallographic technique, followed by sonication in acetone for duration of 10 min. The electrodes were then dipped with deionised water and dried at room temperature. Moreover, 1 M NiSo₄ solution was used as an electrolyte by dissolving analytical grade NiSo₄7H₂O (16.14 g) in 250 cm³ of deionized water.

Electrodeposition was performed using an electrochemical cell or hull cell. The deposition conducted in cell with current variant and at ambient temperature. Ni Electrodeposits were developed by applying direct current of 16mA.cm⁻², 24mA.cm⁻², 32mA.cm⁻² or 40mA.cm⁻² was applied for 20 min. All Electrodeposition experiments were constructed at ambient temperature 20 ±1°C. After the deposition, the samples were dipped with acetone and deionised water before drying at room temperature.

The weight of the Ni deposits was calculated by examining the samples before and after the deposition process. The current efficiency of the Electrodeposition was then computed from weight ratio of Nickel deposits to the theoretically known weight of Ni deposits, which is based on the quantity of current used according to Faraday's laws of electrolysis:

$$M = \frac{QM}{nF}$$

Where m is the mass of the deposits, F is the Faraday constant (96500 C mol⁻¹), Q is the electric charge passed, M is the molar mass of the species, and n is the electrical charge involved in reaction.

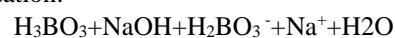
Field Emission Scanning Electron Microscopy (FESEM) was carried out to characterize the morphologies of the Ni electrodeposits. X- ray diffraction (XRD) was also performed on the optimum Ni deposit sample to identify the purity of the sample.

5. Results and Discussions

A. Electrochemical study

Fig. 2, shows voltammogram realized in electrolyte 1. We recognize that the current density is fully stable between -0.3V & -1.3V vs. Ag/AgCl. No current was seen until the potential values greater than -1.3V, then, the cathodic current begins to rise rapidly, which is associated with hydrogen reduction according to: 2 H₂O + 2e⁻ → H₂ + 2OH⁻

The voltammogram realized in a solution containing KCl+H₃BO₃ has the similar shape as electrolyte 1. Boric acid is considered as a very weak acid, with a tabulated ionization constant around pK_a=9.2. At, in the presence of a strong base like NaOH (pK_a=14.8), a reaction takes place according to the following equation:



Hence, boric acid does not have any reaction on the hydrogen evaluation reaction

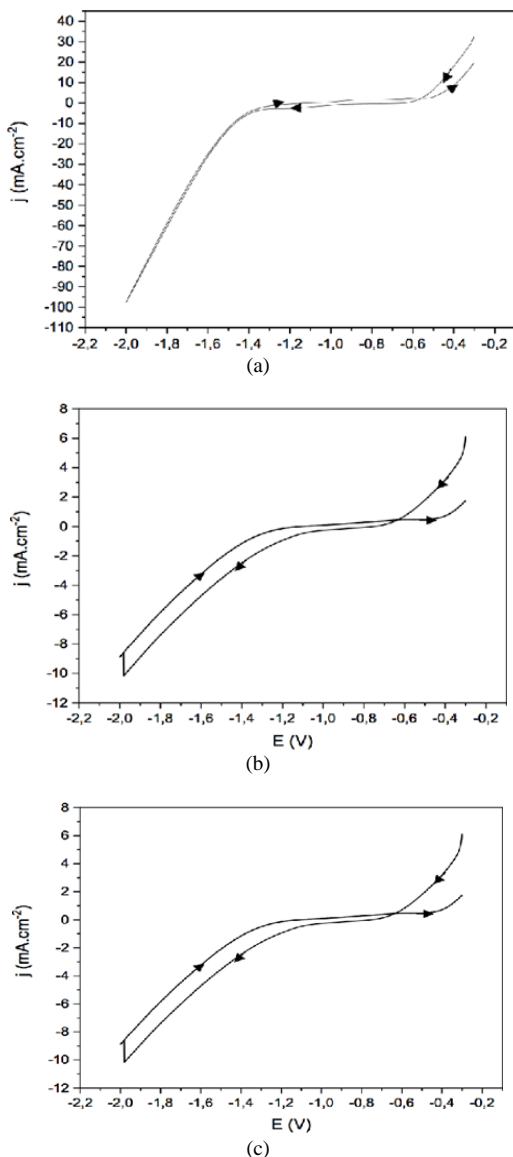


Fig. 2. Cyclic voltammogram on a Fe substrate in (a) KCl solution at pH=4.2, (b) KCl+H3BO3 solution at pH=4.5, (c) KCl+H3BO3 solution at pH=4.5 Scanrate=25mV/s.

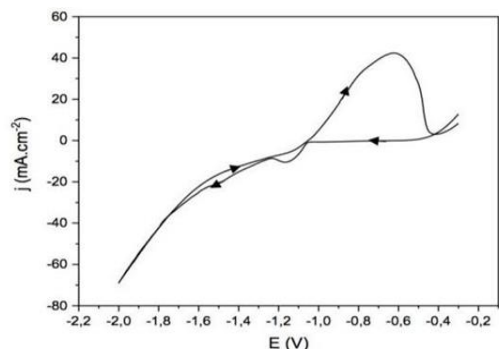
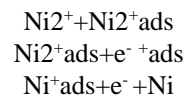


Fig. 3. Cyclic voltammogram of a mild steel in electrolytic bath containing Ni, Scan rate=25mV/s

In Fig. 3, the cathodic current rises instantaneously at -1.05V vs. Ag/AgCl and gives rise to cathodic peak at around -1.17V vs. Ag/AgCl. This rise is related to Ni²⁺-reduction during the cathodic scan according to below reaction:



At about -1.55V, we perceive inflexion; it is mostly due to the formation of the in the preference to the hydrogen. In fact, with current density, Ni displaces Hydrogen already on the surface, reducing the thermodynamically favored hydrogen reaction.

When the potential scan is altered hysteresis appears. Mostly this is crossover potential at which nucleation and growth happens. During the opposite anodic scan; an oxidation rise appears, at -0.6V, which represents the anodic dissolution of the earlier deposited Ni metal.

B. Galvanostatic deposition

Effect of current density the plating bath on deposition potential, thickness, deposition rate and current efficiency Electrodeposition can be made either with the potential or current. In case of the industrial coatings, preparation, the current step method, also known as the Galvanostatic method, is the most experimental. The importance of the Galvanostatic method, is the most experimental. The importance of the Galvanostatic method is that the thickness of the as- deposited layer can be easily modified as per the Faradays law. Accordingly, deposition of Ni coating can be applied using different current density. But also even the applied current density should be superior to the limiting current density of the Ni deposition, which is $\approx -10 \text{ mA} \cdot \text{cm}^{-2}$ as can be observed in Ni voltammogram shown above (Fig. 4). Galvanostatic experiments were carried out in a range of current density varying from 16 to $40 \text{ mA} \cdot \text{cm}^{-2}$.

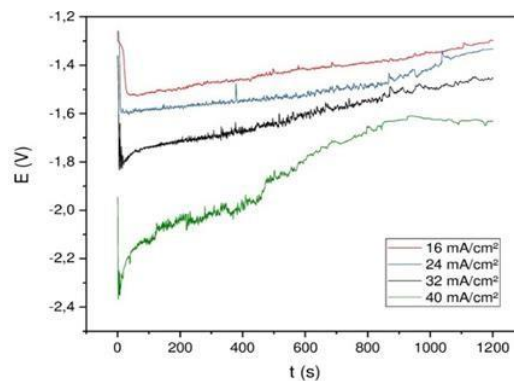


Fig. 4. The evolution of the deposition potential E during Ni Electrodeposition with different current densities

Fig. 4, shows the variation of the deposition of the potential (E) at the time of Electrodeposition with different current

densities. A further increase in current density leads to a notable shift of E_d towards more negative values. For instance, Ni deposition at $16\text{mA}\cdot\text{cm}^{-2}$, $24\text{mA}\cdot\text{cm}^{-2}$, $32\text{mA}\cdot\text{cm}^{-2}$ and $40\text{mA}\cdot\text{cm}^{-2}$ shows an average deposition potential E_d of -1.42V , -1.51V , -1.62V and -1.85V linearly. As increase of the current density leads to stronger potential oscillations in the curves due to Intensive hydrogen evolution.

The impact of current density on thickness (e), which is given by below relation:

$$e = \Delta m / \rho S m / \rho S S$$

where $\Delta m / \rho S m (g)$; the deposited mass of coating; $\rho S (g\cdot\text{cm}^{-3})$; the density of coating and $S(\text{cm}^2)$; the surface are of the substrate, is given in Table 1, it can be observed that the thickness increases with the increasing density, as well as deposition rate $v(\mu\text{m}\cdot\text{h}^{-1})$ which is calculated according to the below equation;

$$v = m_2 - m_1 / \rho S S t$$

Where $m_2(g)$; mass of the samples after deposition, $m_1(g)$; mass of the sample before the deposition, and $t(s)$; deposition time.

Table 1
Effect of current density on thickness, deposition rate and current efficiency

Current density (j) $\text{mA}\cdot\text{cm}^{-2}$	Thickness (e) μm	Deposited rate (v) $\mu\text{m}\cdot\text{h}^{-1}$	Current efficiency (CE) %
16	5.4	17.1	63
24	6.4	19.0	46.8
32	8.3	25.9	46.29
40	11.4	35.3	52.4

Moreover, it is observed (Table 1) that the current efficiency (CE) reduces with rising current density that could be attributed to the rapid rise in hydrogen evolution. The CE is observed using the equation:

$$CE = (\Delta m / \rho S m / m t h) \times 100 \quad m t h = (j t M / n F)$$

(by Faraday's law)

Where $J (\text{mA}\cdot\text{cm}^{-2})$: the applied current density and $M (g\cdot\text{mol}^{-1})$: molar mass of the substance.

Since, in Electrodeposition Experiment Hydrogen is the second element produced at the cathode, many research had investigated the influence of hydrogen evolution on the reaction of Nickel deposition and based on the composition of the electrolyte and plating condition: the current efficiency may differ over wide range. For the evaluation of the impact of hydrogen evolution reaction (HER) on the current efficiency, Dundker et al. have measured the current efficiency by the ratio of the Nickel reaction current density to the whole electrode

current density by below equation:

$$CE \approx \eta_j = (j_{Ni} / j)$$

Where j_{Ni} is the density of the Nickel reaction and j is the total electrode current density. Considering,

$$j = j_{Ni} + j_{H_2}$$

Where j_{H_2} is the current density of the hydrogen reaction. Fig 5.4 represents the variation of polarization of the Ni electrode, in case of considering the impact of HER (j_x : $x=Ni$ or H_2) while in case of not considering its effect ($j_x \setminus H_2$), with different varying current densities.

By not considering the HER effect, we note that Nickel reaction is under mass transfer limitation, which is shown by the observance of the plateau of the Nickel limiting current density. Nevertheless, considering the effect on the Nickel reaction, we bifurcate a significant run of the HER near the mass transfer limitation of the Nickel deposition, showing the improvement of the mass transfer rate of the Nickel ions with hydrogen evolution.

6. Conclusion

In this study nickel coating by electrodeposition method from a simple free additives bath on mild steel. The impact of the current density on the deposition potential are thickness of the deposit, deposition rate and current efficiency was absorbed. The process of nickel will be quite slow and having high corrosion resistance and long durability.

From SEM analysis it is conducted that,

- The Ni coated thin mild steel surface is uniform, bright, crack free and void free.
- The XRD pattern of Ni coated mild steel shows the nano crystalline phase and the average crystalline size was conducted around 83nm.
- The Ni thin layer implies the surface properties of mild steel, thus decreasing its wear rate or increasing wear resistance and also decreases the co-efficient of friction and frictional force.
- Therefore, the uses of Ni thin layer on MS material improves the failure time the material due to wear which plays a huge role in all mechanical feature.

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