# CHARACTERIZATION OF ELECTROLESS DUPLEX NICKEL COATED MILD STEEL

**Abstract**

A variety of industries have high demand of nickel coated materials useful in structural applications. Electroless coating is the autocatalytic deposition of alloy from an aqueous solution on a substrate without the application of electric current. The electroless method has proven to be highly cost effective and flexible process in developing plethora of coating depositions of varying depths. Such coatings can further undergo mechanical treatments to result substantial improvements, useful in particular applications. In the present work the Ni-P, Ni-B and duplex Ni-P/Ni-B coatings have been prepared on mild steel specimen through electro less deposition techniques and their tribological properties have been investigated. A tribometer has been used to estimate the frictional characteristics of the coated samples to compare the friction and wear characteristics of the single and duplex coated elements.

**Keywords:** Electroless coating, Nickel coating, Ni-P coating, Ni-B coating, Duplex coatings

**Authors**

**Sandip Ghosh**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.
sandip.ghosh@jiscollege.ac.in

**Suraj Gazi**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.

**Allen Vivian Lakra**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.

**Suvankar Debnath**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.

**Santanu Ghosh**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.

**Abir Mukherjee**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.

**Palash Biswas**
Department of Mechanical Engineering
JIS College of Engineering
Kalyani, India.

## I. INTRODUCTION

Since the nineteenth century there has been a revolutionary development in the field of Electroless deposition techniques. various subcategories of this Electroless coatings techniques include metallic coatings, alloy coatings and composite coatings [1]. In the alloy coating category the Electroless nickel coating has emerged as a favourable option within many other alternatives due to its excellent results regarding the coated surface properties like wear resistance, corrosion resistance and moderate durability [2]. Generally nickel with Phosphorus or Boron elements are deposited on a substrate through an intricate ionic reduction process in the presence of some reducing agents like sodium borohydride, sodium hypophosphite, etc within a solution bath [3]. To be specific, Ni-P coating can be described as an even coating of nickel-phosphorus alloy which is chemically deposited on the surface of metallic substrate. Investigations reported in literature have shown that the percentage of phosphorus in the alloy can affect its metallurgical characteristics. While the Low-phosphorus-coatings containing up to 4% P contents exhibits mild hardness, High-phosphorus-coatings can have 10–14% P elements. High Phosphorus coatings are are useful in highly corrosive environments like oil drilling as well as coal mining.

Medium-phosphorus coatings with 4-10% P are considered as most common for engineering applications like decorative, industrial and electronic applications [4]. According to the functional application type, the % range of phosphorus varies in the concerned coating process. The reducing agents also have considerable influence on the mechanical characteristics of coated products [5]. Ni-P coatings find several applications while corrosion protection is a primary requisite. At the same time Ni-B coatings demonstrate greater adhesion and higher wear resistance [6]. Sodium borohydride shows better reduction efficiency than dimethyl amino borane popular in Ni-B coating process [7]. In the electrical industry the use of gold can be replaced by electroless Ni-B coatings due to better wear-resistant than tool steels [8]. Although several experimental works have demonstrated the mechanical behaviour of Singular and Duplex Electroless coatings, there is still many applications where appropriate coatings are required and variety of deposition time as well as post deposition treatments needs to be explored. In the present work, several mild steel samples were coated with NiP and NiB deposition in electroless coating methods. Duplex coating were produced by Ni-P/Ni-B and Ni-B/Ni-P deposition methods. Tribological properties like wear rate and coefficient of friction of the coated samples were measured through experimental techniques.

## II. EXPERIMENTAL PROCEDURE

### 1. Coating Deposition

Mild steel pin samples of 30 mm length and 6 mm diameter were used as the substrate for deposition of coatings. A series of processes are involved in the electroless Ni-P or Ni-B coating techniques. First of all 200 ml distilled water is taken in a beaker with the magnetic bar put inside. The Ni-P bath composition includes Nickel Chloride and nickel sulphate both 20 gm/l as the source of nickel and Sodium hypophosphite 24gm/l as the source of phosphorus as a reducing agent. Sodium succinate of 12gm/l as stabilizer is added in the solution. [9]. For Ni-B bath Nickel chloride 25 gm/l, sodium borohydrate 1gm/l and lead nitrate as per appropriate measurement is applied. Once the solutions are ready, mild steel samples are placed in their corresponding bath and kept at a temperature of 82 degree

Celsius. For each layer of coating the coating process was continued for 1 hrs of time. In the Table 1 and Table 2, the compositions of Ni-P as well as Ni-B bath are depicted respectively.

**Table 1:** Composition and Conditions of Ni-P bath [1]

| Sl. No | Component and condition | Quantity |
|---|---|---|
| 1 | Nickel sulphate (g/l) | 25 |
| 2 | Nickel chloride (g/l) | 25 |
| 3 | Sodium Succinate (g/l) | 12 |
| 4 | Sodium hypophosphite (g/l) | 24 |
| 5 | Temperature ($^oC$) | 90 ± 2 |
| 6 | pH | 12.5 |

**Table 2:** Composition and Conditions of Ni-B bath [1]

| Sl. No | Component and condition | Quantity |
|---|---|---|
| 1 | Nickel chloride (g/l) | 20 |
| 2 | Sodium borohydride(g/l) | 0.8 |
| 3 | Ethylenediamine(g/l) | 59 |
| 4 | Lead nitrate(g/l) | 0.0145 |
| 5 | Sodium hydroxide(g/l) | 40 |
| 6 | pH | 12.5 |
| 7 | Temperature ($^oC$) | 90 ± 2 |

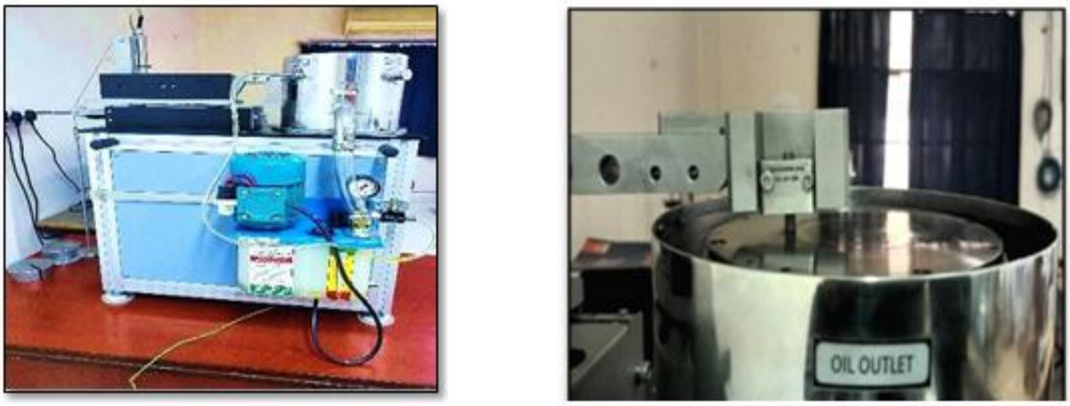
**Figure 1:** NiP (left) and NiB (right) coating solution bath

## 2. Microhardness Study

In the present investigation, samples undergo a micro-hardness test in a Vickers tester (UHL-VMHT) using an indentation load of 100 g-f at a rate of 25 μm/s for a dwell period of 15 s. The samples are indented using a diamond indenter with a square base. Optical microscopy is used to measure the indentation size. Because of the thin coating, a lower indentation load value was used. Hardness and wear resistance are measured three times, with the average value being reported.

### 3. Tribological Study

Pin-on disc Tribometer is highly popular in examining the friction and wear properties of the EN-coated specimens. The same was used in the present work (Fig. 2). Firstly, the coated samples were tested in dry condition at room temperature. The tribometer typically uses a load cell along with a linear variable differential transformer to measure the frictional force and resulting displacements of pin. The disc is rotated by A variable-speed motor rotates the disc while preventing any vibration affecting the test. The normal load placed on the disc through the pin in the current work is 25 N. The disc's track diameter is kept fixed at 70mm, and its spinning speed is 60 RPM in reference to several reported works in literature [9, 10, 11]




(a) (b)

**Figure 2:** (a) Pin-On-Disc Tribometer, (b) loaded pin setup during test

### 4. Microstructural Observation

In the last phase of the work, Scanning Electron Micrograph (JSM 6360, JEOL) has been used to examine the produced coatings' surfaces morphology and its characterization.

## III. RESULT AND DISCUSSION

### 1. Morphology of the Coated Surface

In the Fig. 3 the SEM micrography of the as-deposited coated samples have been presented. In the Ni-P coated sample, nodular structure can be observed with an average nodule size of 10-15 µm. No porosity is found in the SEM image fig 3(a). For Ni-B coating bigger cauliflower-like nodule structure can be seen (Fig.3(b). For both the duplex coating relatively, smoother surface can be observed. Such structure is suitable for better hardness and less frictional resistance.

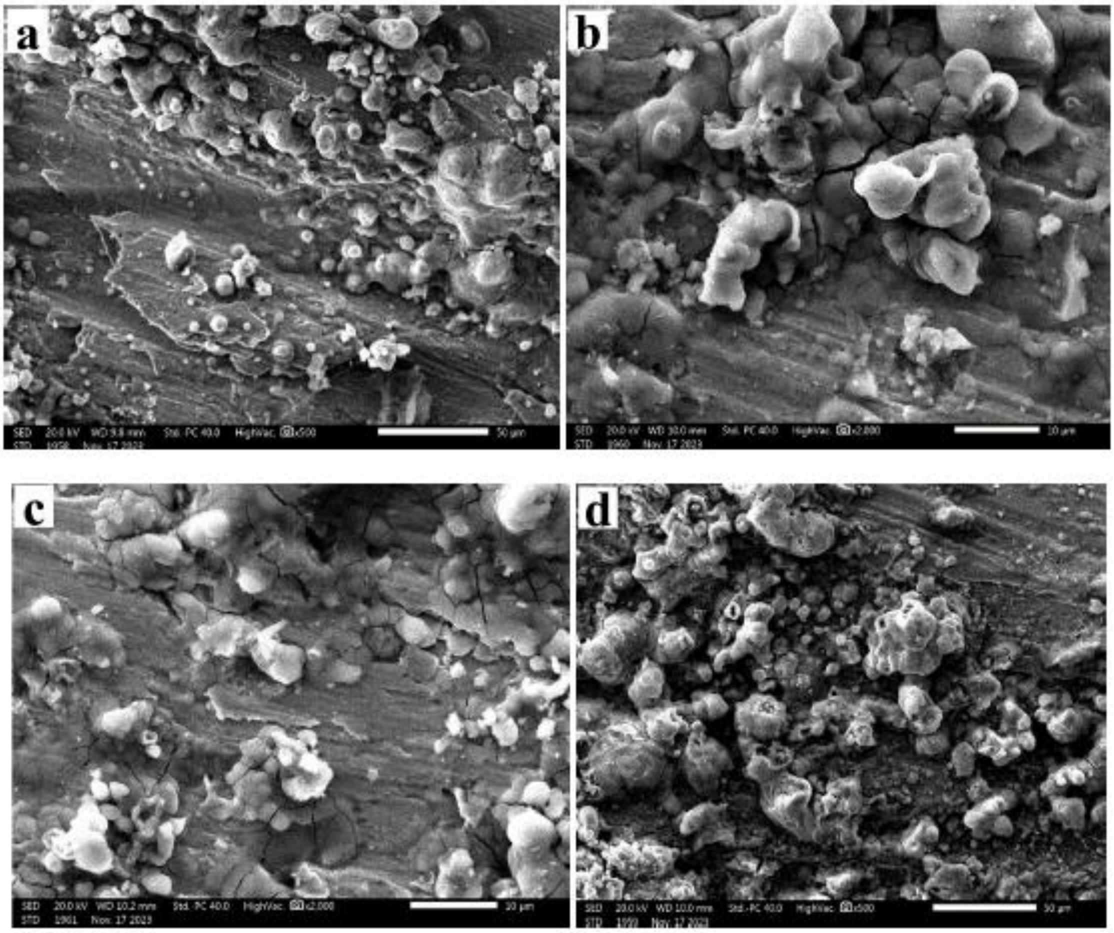



**Figure 3:** Microstructure of electroless (a) Ni-P coating (b) Ni-B coating (c) duplex Ni-P/Ni-B coating and (d) duplex Ni-B/Ni-P coating

## 2. Microhardness Estimation of Electroless Duplex Nickel Coating

Duplex electroless nickel coatings are shown to have better hardness values, and electroless nickel coatings are well known for their great hardness. As illustrated in Fig. 4, duplex electroless Ni-P/Ni-B coating is found to have greater hardness than other electroless nickel coating in the as-deposited condition due to the harder phase on the outer layer. For Ni-B coating is obtained higher hardness values than electroless Ni-P single layer or duplex Ni-B/Ni-P coating. In Ni-B deposits, boron atoms integrate within the nickel matrix, forming intermetallic compounds like nickel borides. These nickel boride particles are significantly harder than the nickel phosphide that forms in Ni-P coatings. Because of the smaller phosphorous concentration of the microcrystalline deposit, Ni-P/Ni-B coating's hardness values are around 17% higher than duplex Ni- B/Ni-P coating.

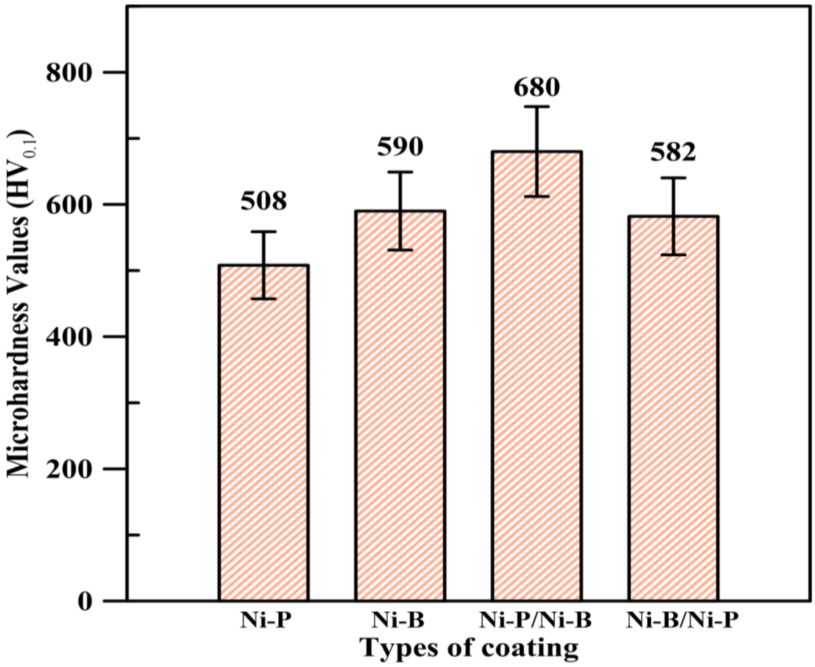



**Figure 4:** Microhardness study of various coating

## 3. Friction Performance

Friction performance of duplex electroless nickel coatings is naturally smooth. Fig. 5 shows the coefficient of friction (COF) plot for several coating types. Because duplex electroless Ni-P/Ni-B coating's surface is less smooth than that of the other coatings, its COF is higher. Because of the top layer in the duplex Ni-P/Ni- B coating is a harder material (e.g., Ni-B), it might generate higher friction against the contacting surface, leading to a higher COF. This is because the harder layer offers more resistance to movement during sliding contact. COF considerably decreases by around 25% for duplex Ni-B/Ni-P coating compared to Ni-P/Ni-B coating. Additionally, COF for the Ni-P coating is minorly lower than for Ni-B coating due to the soft surface. For polycrystalline materials, the duplex Ni–B/Ni–P exhibits the lowest COF. The efficiency of friction is influenced by grain size. The minimal COF of duplex Ni-B/Ni-P coating was around 0.48. The creation of distinct phases, the distribution of phases, and other aspects all affect how the coatings behave when it comes to friction.

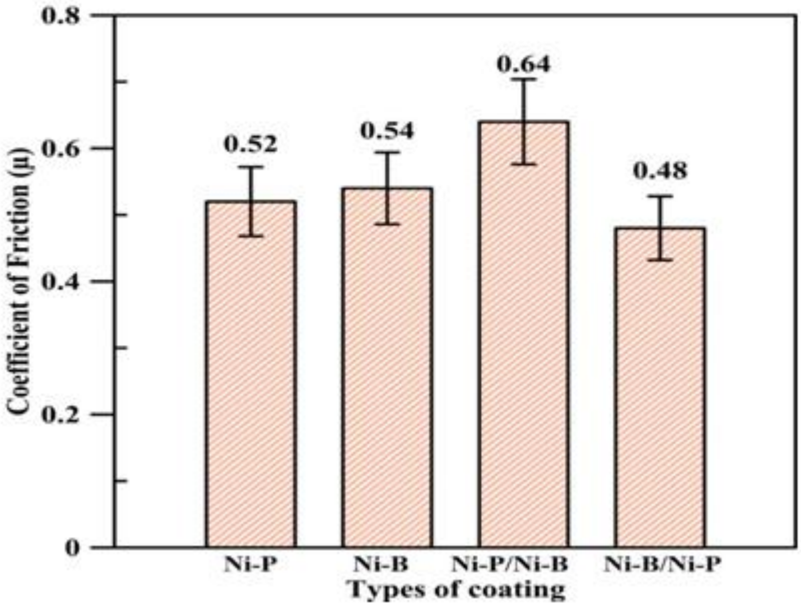



**Figure 5:** Friction performance of various electroless coating

## 4. Wear Behaviour

Fig. 6 shows the results of wear testing done on several coated sample types. Because Ni–B coating is present in the outer layer, Ni-P/Ni-B coating has the lowest wear rate. For coating Ni-B/Ni-P, where Ni–P makes up the outer layer, the wear rate is higher than duplex Ni-P/Ni-B coating. Duplex Ni-P/Ni-B coating showed the least wear and maximum hardness. For duplex Ni-P/Ni-B coating, Figures 4 and 6 demonstrate improved hardness and a reduced wear rate with a strong connection. Duplex coatings combine the beneficial properties of two different layers. A common approach is to use a hard layer. This layer, often nickel-boron (Ni-B), provides excellent wear resistance due to its high hardness and the presence of hard nickel boride particles. It acts like a shield, taking the brunt of the wear and tear from contact. The interface between the two layers can create a gradual transition in properties, reducing stress concentrations and potential crack initiation points that could lead to wear. For both the duplex coating has obtained a lower wear rate than consecutive successive single layer coating. Duplex coatings can offer a combination of properties like high hardness, good toughness, and improved load-bearing capacity compared to single-layer coatings. This can help them resist wear under various contact conditions.

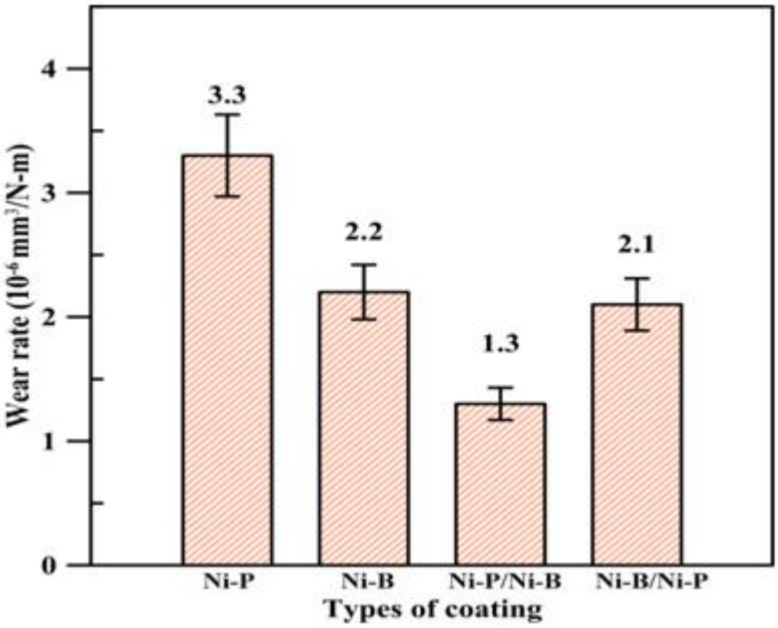



**Figure 6:** Wear rate of various electroless coating

## IV. CONCLUSION

By successively dipping inside two coating solution baths, the current investigation demonstrates that using the electroless single-layer Ni-P & Ni-B coating, duplex Ni-P/Ni-B, and Ni-B/Ni-P coatings may be prepared. Temperature variations appear to cause a small breakdown of the Ni–P solution. For two hours, the Ni-B bath is shown to be significantly more stable without bath breakdown.

- From a microstructural perspective, duplex coatings exhibit homogeneous coatings and good interlayer compatibility about grain boundaries and nodular development.
- Better wear resistance can be achieved in duplex Ni-P/Ni-B coating in comparison with others.
- When considering all coating types combined, single-layer Ni-P coating has the lowest hardness. The duplex Ni–P/Ni–B has the highest value of hardness. When it comes to hardness and wear resistance, duplex coating is the most effective type of coating out there.

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