Comparative Tribological Characterization of Electroless Ni–P, Ni–B, and Duplex Ni–P/Ni–B Coatings on Mild Steel

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Abstract: A variety of industries have a high demand for nickel-coated materials useful in structural applications. The electroless method has proven to be a highly cost-effective and flexible process in developing a plethora of coating depositions of varying depths. Such coatings can further undergo mechanical treatments to result in substantial improvements useful in specific industrial applications. In the present work, the Ni-P, Ni-B and duplex Ni-P/Ni-B coatings have been prepared on mild steel specimens through electroless deposition techniques, and their tribological characters 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. It was observed that the duplex Ni-P-Ni-B coated samples exhibits 60% less wear while NiB-NiP coated samples showed significantly higher microhardness among all three variants.

Keywords: *Coating, Friction, Electroless, Duplex, Nickel, Tribology.*

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I. INTRODUCTION

Electroless coating is the autocatalytic deposition of alloy from an aqueous solution on a substrate without the application of electric current. Since the nineteenth century, there has been a revolutionary development in the field of Electroless deposition techniques. Various subcategories of these 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 is 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 a metallic substrate. Investigations reported in the 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 content exhibit mild hardness, high-phosphorus coatings can have 10–14% P content. High-phosphorus coatings 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 the 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 resistance than tool steels [8]. Although several experimental works have demonstrated the mechanical behaviour of Singular and Duplex Electroless coatings, there are still many applications where appropriate coatings are required and a variety of deposition times as well as post-deposition treatments need to

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be explored. In the present work, several mild steel samples were coated with NiP and NiB deposition in electroless coating methods. Duplex coatings 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. MATERIALS AND EXPERIMENTAL METHOD

- *Coating Deposition*

Mild steel pin samples of 30 mm length and 6 mm diameter were used as the substrate for the deposition of coatings. A series of processes are involved in the electroless Ni-P or Ni-B coating techniques. First of all, 200 ml of 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 stabiliser is added to the solution. [9]. For Ni-B bath, Nickel chloride 25 gm/l, sodium borohydrate 1gm/l and lead nitrate as per appropriate measurement are applied. Lead nitrate is used as a stabilizer to prevent the uncontrolled decomposition of the solution, while ethylenediamine is used as a complexing agent, controlling the concentration of free nickel ions while influencing the plating rate and boron content in the bath. Once the solutions are ready, mild steel samples are placed in their corresponding bath and kept at a temperature of 82 degrees Celsius. For each layer of coating, the coating process was continued for 1 hrs of time. In Tables 1 and 2, the compositions of Ni-P as well as Ni-B baths 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

Fig1 NiP (Left) and NiB (Right) Coating Solution Bath

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➢ *Microhardness Study*

In the present investigation, samples undergo a micro-hardness test in a Vickers tester (UHL-VMHT) using an indentation load of 100 gf 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.

➢ *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 conditions 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 the pin. The disc is rotated by A variable-speed motor that 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, according to several reported works in literature [9, 10]

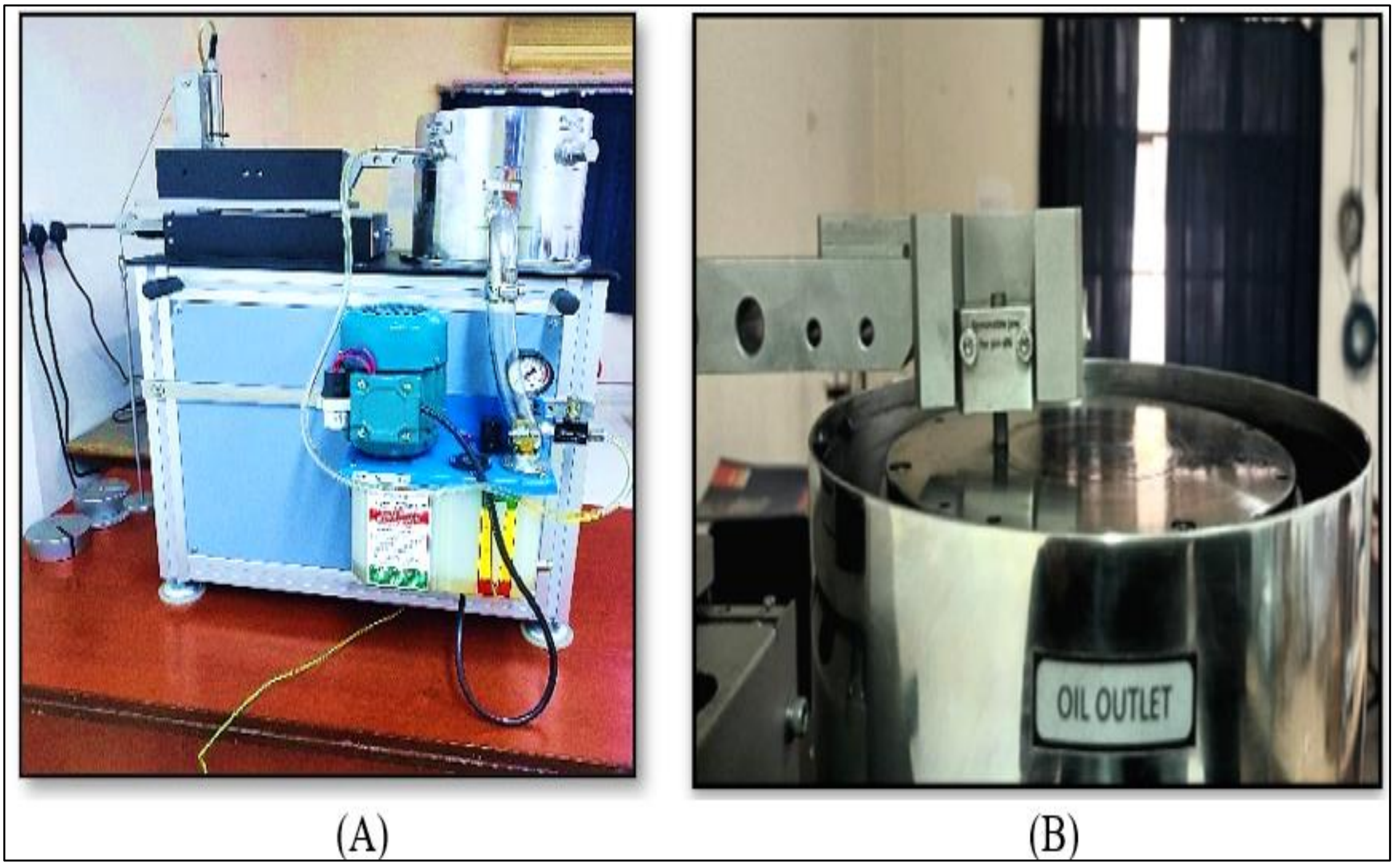

Fig 2 (A) Pin-On-Disc Tribometer, (B) Loaded Pin Setup During Test

➢ *Microstructural Observation*

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

III. RESULTS AND DISCUSSION

➢ *Morphology of the Coated Surface*

In Fig. 3, the SEM micrograph of the as-deposited coated samples is 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, a smoother surface can be observed in the images. Such a structure is suitable for better hardness and less frictional resistance as reported in previous reports [11,12]. Fig. 4 displays EDX spectrum of the present duplex coating along with the element composition in as-deposited conditions. Coatings are classified as high phosphorous materials based on their phosphorus concentration. Also, duplex Ni-P/Ni-B coating observed the Ni and B concentration for the top surface of the coated materials.

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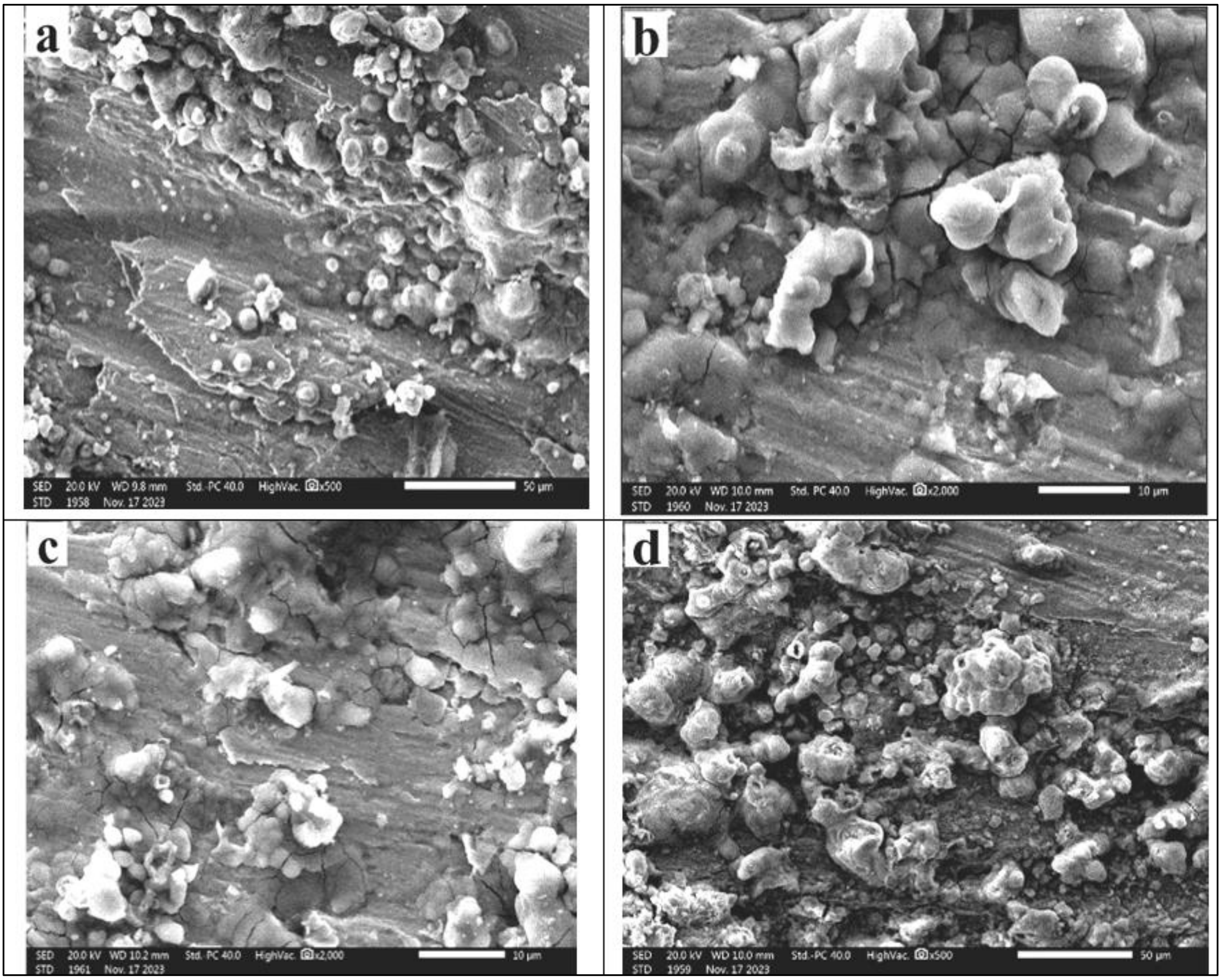
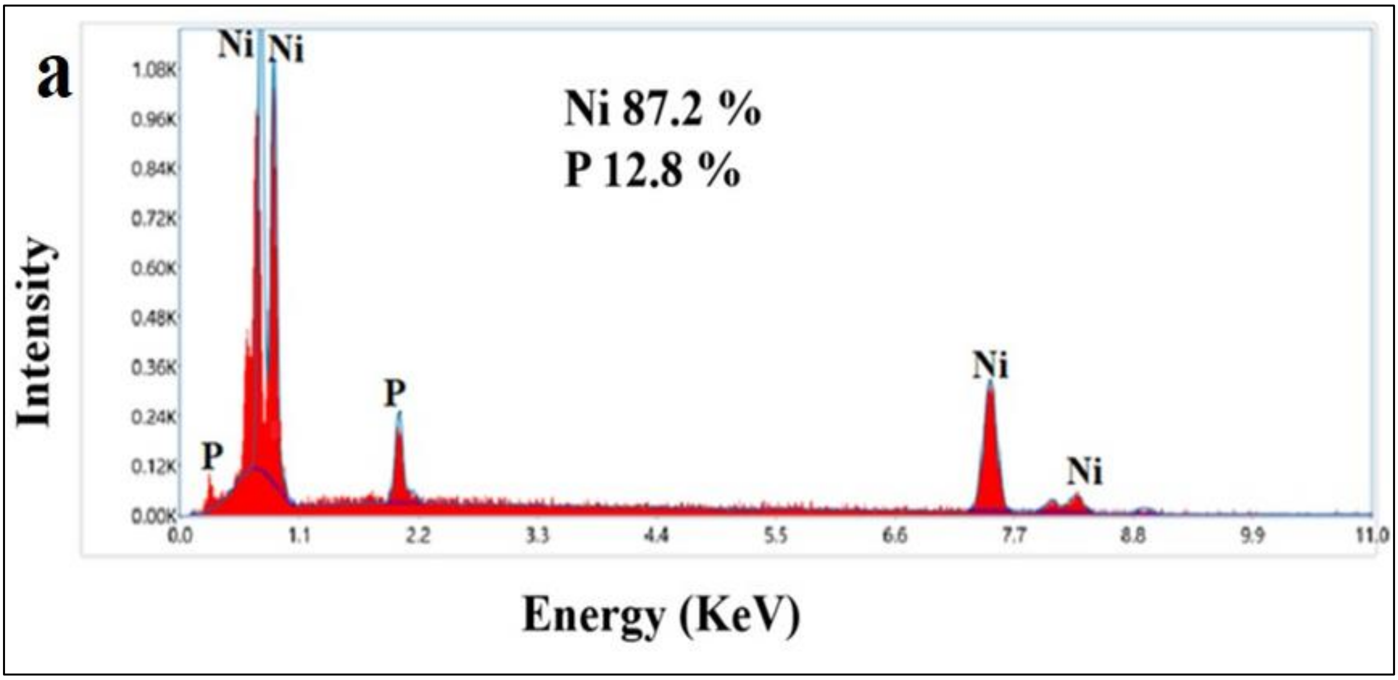


Fig 3 Microstructure of Electroless (a) Ni-P Coating, (b) Ni-B Coating, (c) Duplex Ni-P/Ni-B Coating and (d) Duplex Ni-B/Ni-P Coating



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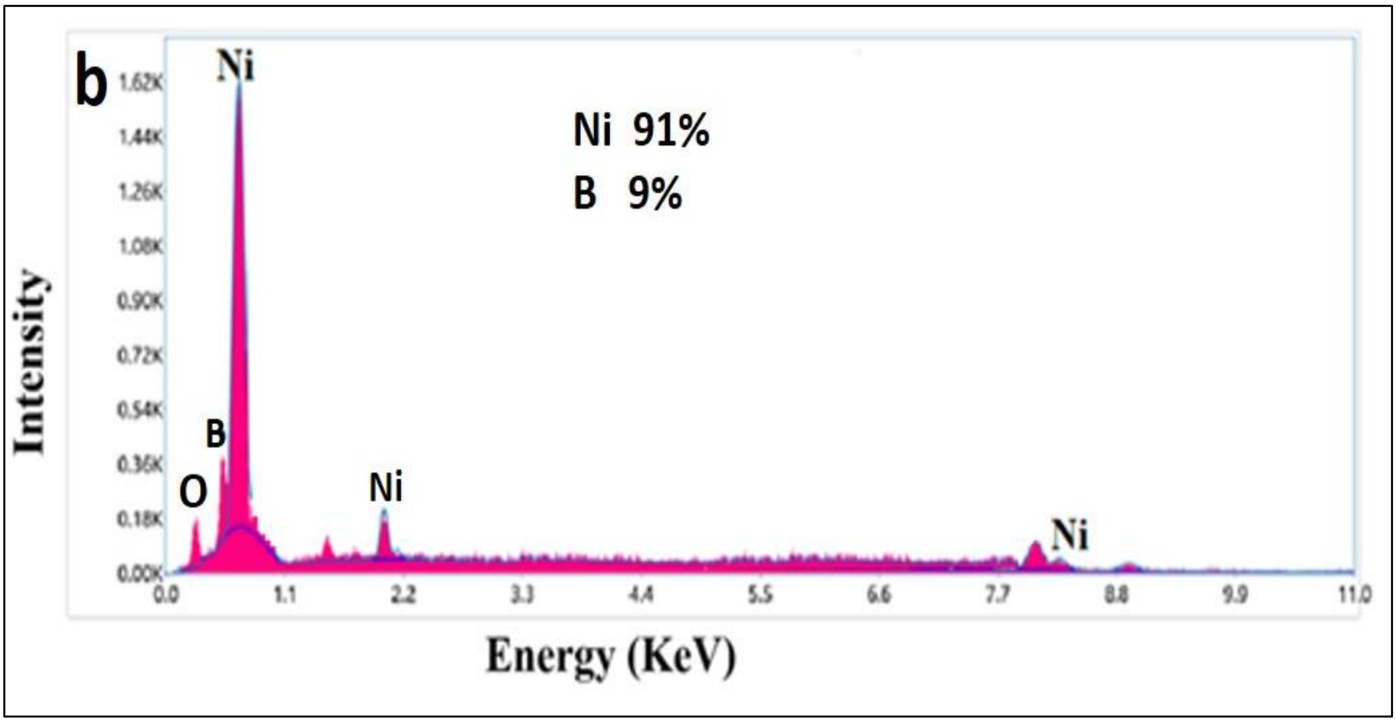

Fig 4 EDX Spectra for as-Deposited Duplex (a) Ni-B/ Ni-P and (b) Ni-P/ Ni-B Coating

➢ *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. 5, duplex electroless Ni-P/Ni-B coating is found to have greater hardness than other electroless nickel coatings in the as-deposited condition due to the harder phase on the outer layer. For Ni-B coating is obtained higher hardness values are obtained 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 phosphorus concentration of the microcrystalline deposit, Ni-P/Ni-B coating's hardness values are around 17% higher than duplex Ni-B/Ni-P coating.

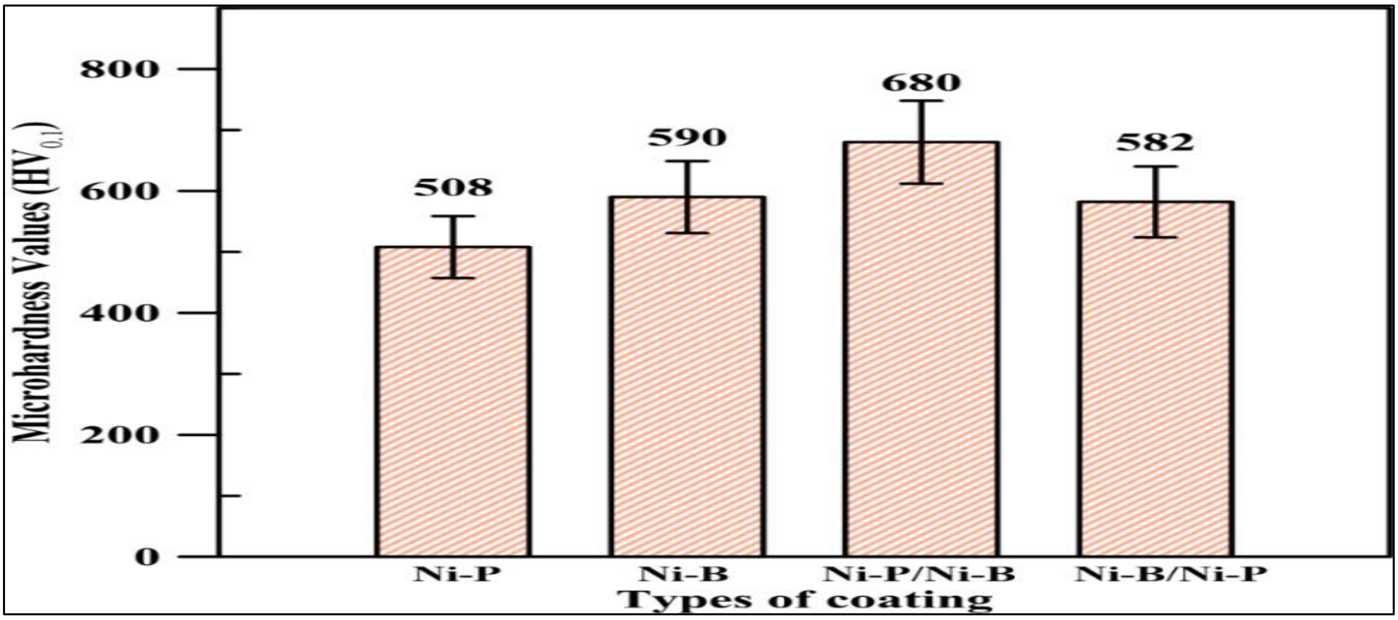

Fig 5 Microhardness Study of Various Coatings

➢ *Friction Performance*

The friction performance of duplex electroless nickel coatings is naturally smooth. Fig. 6 shows the coefficient of friction (COF) plot for several coating types. Because the duplex electroless Ni-P/Ni-B coating's surface is less smooth than that of the other coatings, its COF is higher. Because 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

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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, the COF for the Ni-P coating is slightly lower than for the 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 the 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.

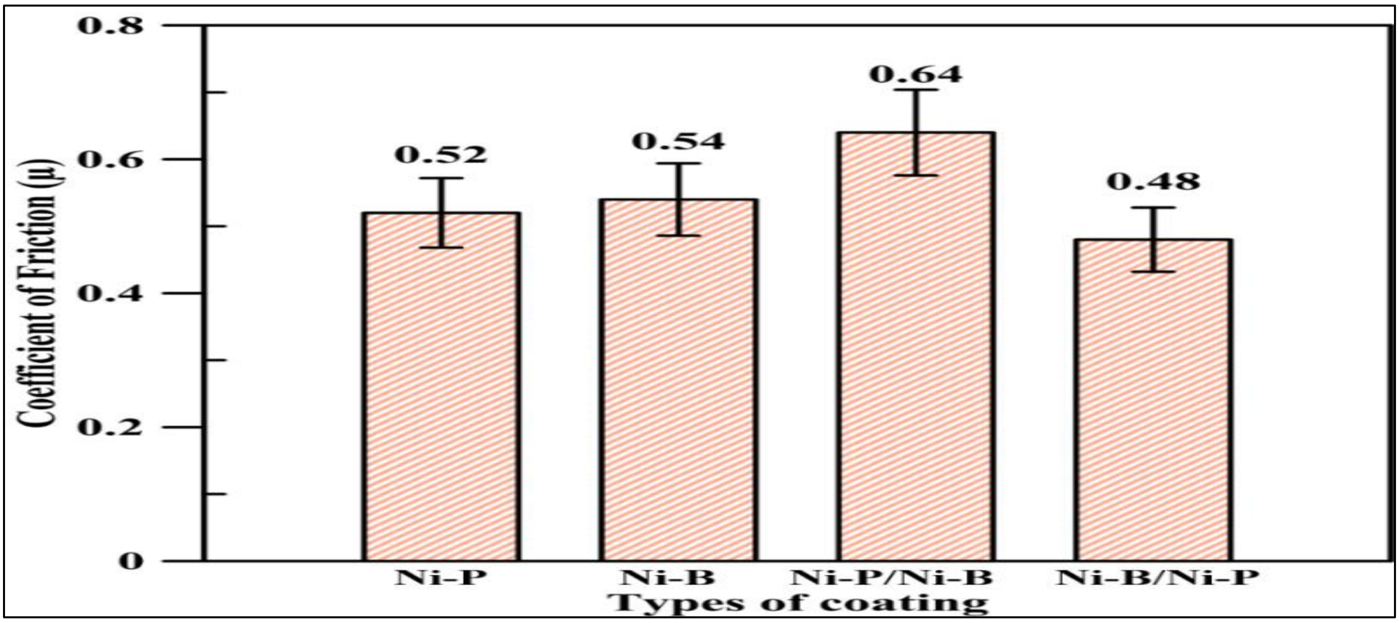

Fig 6 Friction Performance of Various Electroless Coatings

- *Wear Behaviour*

Fig. 7 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 the consecutive 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.

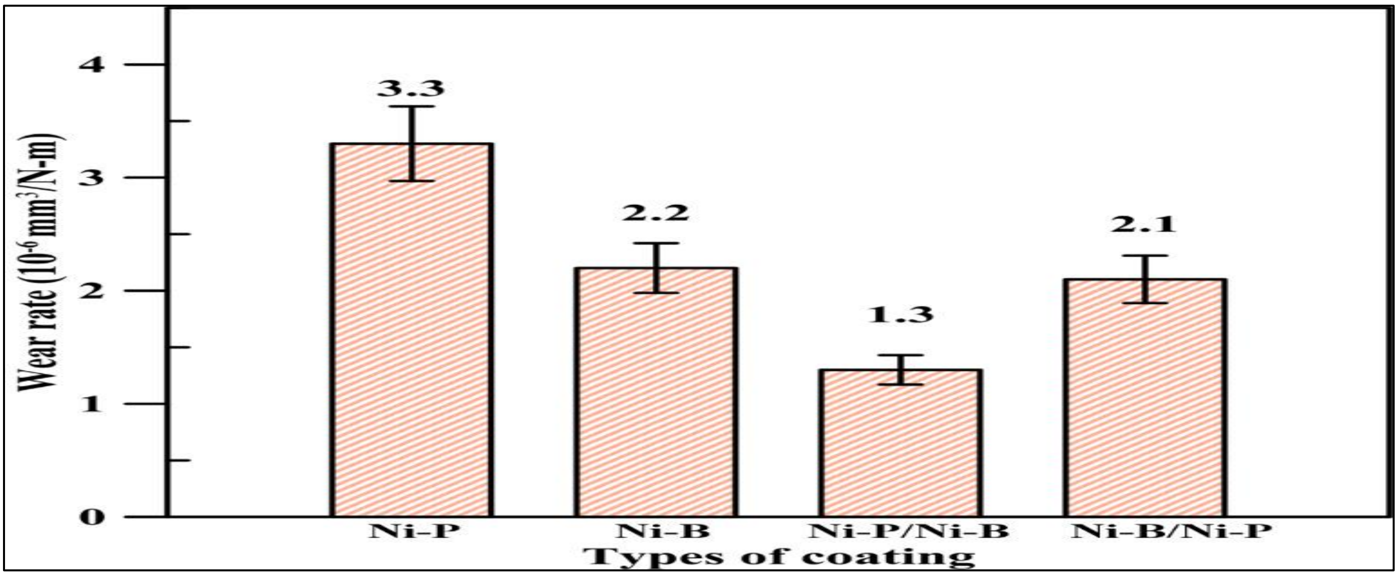

Fig 7 Wear Rate of Various Electroless Coatings

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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 at grain boundaries and nodular development. With almost 60% less wear rate than single layer Ni-P coating, a significantly better wear resistance can be achieved in duplex Ni-P/Ni-B coating. When considering all coating types combined, the has the lowest hardness. The duplex Ni–P/Ni–B showed around 33% higher microhardness in comparison to single-layer Ni-P coating. When it comes to hardness and wear resistance for applications in aerospace, automotive, and electronic industries, duplex coating shows marvellous performance as the most effective type of coating.

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