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## **Experimental Studies of Hardness Value and Corrosion Behavior of Coated Steel Bar**

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Abstract: Now-a-days steel is used in various construction structure such as building, bridges, pavement etc., but the major issue with the steel is, it easily gets corroded. Corrosion causes many important structure damage, many methods are used to increase the corrosion property of steel. Electroless coating was one of the modern technique to modify the surface property so that steel corrosion property. Ni-P coating was applied on steel 304. In this paper, Ni-P concentration variation and temperature variation was done along with presence or absence admixtures (such as-Lactic acid, Succinic acid, Citric acid) were used. The surface morphology was determined by FESEM Analysis and XRD Analysis, but hardness value and corrosion resistance of the substrate was determined by Vicker's micro-Hardness test and EIS Analysis respectively. Results obtained from these tests showed that the coating thickness increases with increases in Ni-P concentration as well as temperature increment in presence of admixture. In micro-Hardness test, hardness value of substrate was improved as coating thickness increases. Electrochemical impedance spectroscopy (EIS) test results also showed that corrosion potential of improved coated substrate was enhanced or modified than previous sample substrate.

Keywords: Morphology, Micro-Hardness, Oxidation, Etching, Desmutting.

## I. INTRODUCTION

Steel bar get corroded when it comes in contact with moisture for a longer contact period. Since the passive film on carbon steel gets degraded after passage of time due to many reasons. These reasons may be due to presence of chloride, insufficient cover, cracks in concrete and due to carbonation process. When the steel bar start corroding, deterioration of concrete starts, As volume gets increased due to corrosion, cracks gets developed in the concrete and thus we loss the structure. Nihal et al. [1] reported that corrosion process alters the external surface of the steel bar due to pitting. The residual section of corroded steel bar is no longer round and varies considerably along its circumference and its length, therefore the residual diameter is better defined by loss of weight. The yield stresses are reduced with an increase of corrosion degree, while the (Es) value did not remarkably change .As volume of corroded steel gets increased and become greater than original one, so structure gets collapsed du e to separation of steel parts, the process is known as rust packing. And if the process occurs in concrete, it will increase the stress inside the concrete finally failure of structure takes place and the process is called, spalling of concrete. The alkaline compounds, mainly calcium and to a certain extent pottasium and sodium, in the cement contribute to the high alkalinity (pH>13.5) of the pore solution. At this high pH, steel is passivated in the presence of oxygen due to the formation of a sub-microscopically thin  $\gamma$ -Fe2O3 film [2,3]. Hime and Erlin [4] suggested that the passivating layers on the steel surface could have a composition other than that of  $\gamma$ -Fe2O3. According to Page [5], the lime-rich layer at the steel concrete interface, provides further protection to the steel. This was confirmed by Leek and Poole [6] who reported that the interfacial layer consists of an aggregates-free zone of portlandite [Ca(OH)2] of variable thickness (5-15µm) disrupted by inclusions of calcium silicate hydrate (C-S-H) gel. The layer is thought to protect the surface of the steel from direct access of aggressive ions and to act as an alkaline buffer to pH reductions resulting from the hydrolysis of corrosion products [7]. According to Sagoe-Crentsil and Glasser [8], both Ca(OH)2 and C-S-H gel form a buffering pair and a high pH is as readily maintained by C-S-H as by Ca(OH)2. Reinforced concrete frames are the most commonly adopted building construction practices in world. Due to the scarcity of land because of rapid growing of population, high-rise structures have become highly preferable in buildings scenario. A natural hazard like Earthquake causes damage or collapse of these high rise structures, if not designed for lateral loads. Hence such high-rise structures, constructed especially in highly seismic prone zones, should be analyzed and designed with extra lateral stiffening system to improve their seismic performance and reduce damages. Two of the most commonly used lateral stiffening systems in buildings are shear walls and bracings. Both have better seismic performance due to improved lateral stiffness and lateral strength.



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### A. Corrosion of Steel Bar

When steel comes in contact with atmospheric moisture it starts deterioration of itself .Since anode and cathode are formed on steel surface so that there redox reaction takes place .In this process iron gets oxidized in its corresponding ferrious and ferric state and it releases electrons, and that electron is gained by oxygen molecule present in atmosphere. Following chemical reaction takes place as  $E_{i} = E_{i}^{2} + 2$ 

$$Fe \rightarrow Fe^{+} + 2e^{-}$$

$$O_{2}+4e^{-} + H2O \rightarrow 4OH^{2}$$

$$4Fe^{2+} + O_{2} \rightarrow Fe^{3+} + O^{-2}$$

$$2 Fe^{3+} + 4OH^{-} \rightarrow 2Fe(OH)_{3}$$

So a rusted layer is formed, hence member gets deteriorated and hence structure. It will not cause only loss of money, it will also cause a huge damage of precious human life.

#### B. Coatings on Bars

In our modern age many methods are used to protect against corrosion of steel structural members. Methods of corrosion control for reinforcing steel include cathodic protection [9,10], surface treatment of rebars (e.g., epoxy coatings), usage of a surface coating on the concrete [11] and the usage of mineral admixture (e.g., silica flume) in the concrete. GQDs as a new class of zero dimensional graphene based materials with sizes below 100 nm (mostly range from 3 to 20 nm) have currently received increasing attention because of their unique physicochemical properties including large surface area, low cytotoxicity, excellent biocompatibility, strong quantum confinement and edge effects [12]. Undeniably, with the purpose of achieving the successful application of GO (Graphene oxide) in the epoxy coating, the main issue to be settled urgently is improving dispersion of GO. One of the approaches to increase GO's dispersion into epoxy coatings is the chemical modification of GO with polymeric solubilizing groups [13,14] The use of advanced epoxy-based materials with superior properties has undergone serious developments in recent years and it is estimated to take a particular place in between versatile thermosetting systems such as nanocomposite coatings [15-17].Our nanocomposite will be a good coating material for corrosion resistivity either against chloride or carbonation.

#### **II. OBJECTIVE OF STUDY**

- A. To improve the resistance of steel 304 substrate against corrosion.
- B. To increase the durability of steel 304 substrate and hence the structure.
- C. To improve wear resistance of steel 304 substrate.
- D. To improve hardness value of steel 304 substrate.

#### **III. LITERATURE REVIEW**

Sahar Afroukhteh et al. [18] studied on the effects of addition of the surfactants on surface morphology, deposition rate, TiC and P contents of the prepared coatings. Sahar demonstrated that in the presence of the anionic, polymeric and somehow cationic surfactants, TiC nano-particles were embedded in the matrix which influenced the surface morphology. The effect of surfactant types on the corrosion properties of Ni–P/TiC coated steel was also studied.







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The TiC incorporation of the coating increase with the addition of surfactant especially in presence of anionic surfactant. The higher concentration of anionic surfactant also increased the TiC incorporation in the coating. Addition of surfactant promoted the corrosion resistance of the Ni–P/0.1 TiC coated steel especially in presence of polymeric surfactant. Inaddition, the corrosion resistance of the coated steel was raised by an increase in anionic surfactant concentration upto 3ml/l.

M. Emamy et al. [19] applied coating on low carbon steel with the incorporation of different amounts of nano Al2O3 powder in electroless bath. The dispersion stability of alumina colloidal particles stabilized by polymeric (non-ionic) surfactants in an electroless bath was also investigated by Sahar.



Fig 2.2 shows Potentiodynamic polarization curves of Ni-P with varying surfactant PEG

The results showed that increasing alumina concentration not only changed the surface morphology, but also enhanced the corrosion resistance. Addition of surfactants has an indirect effect on the amount of the incorporated particles. Sahar investigated that in the presence of surfactant, corrosion resistance of Ni–P coating containing even a small quantity of alumina was improved since a stabilized bath was obtained.

Preeti Makkar et al. [20] studied to incorporate electroless Ni-P-ZrO<sub>2</sub> nanocomposite coatings on mild steel substrate where the reinforced nano-sized ZrO<sub>2</sub> particles were prepared by mechanical milling using high energy planetary ball mill. An alkaline bath was used with a suspension of ZrO<sub>2</sub> particles (4 g/L) for the synthesis of Ni-P-ZrO<sub>2</sub> composite coating. The results showed that  $ZrO_2$  particles exhibit irregular shaped and size ranges from 14 to 17nm. After heat treatment (400°C,1h), the microhardness and wear resistance of the coatings are significantly improved. The Ni-P-ZrO<sub>2</sub> nanocomposite coatings exhibit enhanced wear resistance over Ni–P coating.

T Radu et al. [21] shows that Co-deposition of  $Al_2O_3$  particles in electroless Ni–P coating and their influence on the coating layer characteristics depend on their size, concentration in the electrolyte, by the duration of the deposition process and the way to maintain good dispersion of particles in electroless Ni–P bath. The paper analyzes the effect of nanoparticles (20nm) and micrometer (0,3µm) of  $Al_2O_3$  incorporated in the electrolyte in amounts of 3g/L, 5g/L and 8g/L on the different characteristics of the composite coating layer. The corrosion resistance has been found increasing for increasing alumina content both for micrometric and nanometric alumina.

Zhiming gao et al.[22] investigated on CNTs intersperse in the gaps between the deposited metal particles and the size of the metal particles is much smaller than the size of the metal particles which aren't wrapped with CNTs. The codeposition of Ni-P-CNTs can work as a kind of lubricant between the coating and the other friction body. Compared to the Ni-P coating, the wear resistance and the surface hardness value of Ni-P-CNTs coating are better, and they become even better with the increase in the CNTs concentration. The addition of CNTs makes the coating denser, thus compared with Ni-P coating, the corrosion resistance of composite coating is improved. But too much CNTs deposited on the substrate occupy the position of the nickel and phosphorus, leading to a large area of the carbon nanotubes wrapped together which makes it very unfavorable to coating, resulting in the



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corrosion potential of the composite coating becoming negative gradually, indicating the corrosion resistance of the composite coating becomes worse. It achieves the best value when the CNTs concentration was 0.5 g/L.

Sinem Eraslan et al. [23] have studied on oxidation behavior of electroless Ni–P, Ni–B and Ni–W–B coatings deposited on steel substrates. High temperature oxidation behavior of electroless Ni–P, Ni–B and Ni–W–B coatings which are deposited on mild steel substrate, investigated in a temperature range of 600–800 °C. Oxide films formed on the surfaces of the coated mild steel samples were characterized by X-ray diffraction analyses. Oxidation behaviors of the coatings were evaluated by monitoring the oxidation character and iron diffusion through the structure by glow discharge optical emission spectroscopy (GDOES). The results indicated that oxidation properties of the coated steel depend not only on the coating itself, but also on the diffusion and interdiffusion processes, which take place between the substrate and the deposit. For Ni–P deposited steel, iron started to take part in the oxide structure during treatment at 700 °C, resulting in a loss of protective character of the oxide. Depletion of boron within the coating and formation of nickel borates were observed for B containing deposits. Diffusion of iron to the surface also took place at 700 °C for Ni–B coatings, but not as drastic as for Ni–P. Oxidation behavior of Ni–W–B coatings was similar to Ni–B coatings in general. However, by increasing W, decreasing the diffusion of iron to the surface and delaying the formation of iron oxides. At 800 °C, all the three coatings started to degrade due to their higher oxidation rate and iron diffused to the surface. The results of this study showed that it is possible to increase the oxidation resistance of electroless nickel coatings by alloying them with W.

A.S. Hamada et al.[24] investigated on indentation property and corrosion resistance of electroless nickel-phosphorus coatings deposited on Austenitic High-Mn TWIP Steel bar. A multilayer coating using electroless nickel-phosphorus (Ni-P) applied on a twinning-induced plasticity (TWIP) steel containing nominally 25 wt.% Mn and 3 wt.% Al to improve the indentation hardness and corrosion properties. Micro indentation tests with two different indenters, namely, a three-sided pyramidal Berkovich indenter and a ball indenter were performed to study the mechanical response, the indentation hardness and elastic modulus of the coatings in conditions: as-plated, and post treated (PT) at 350 °C and 700 °C for 1 h. The deformation morphology underneath the indenters was examined using a scanning laser microscope. The results showed that Ni-P coatings could significantly enhance the surface hardness of the TWIP steel. Hamada showed that significant improvement in the corrosion resistance could be observed in a sulfuric acid solution for the Ni–P coated steel compared to the uncoated substrate TWIP steel.

Junning Li et al. [25] investigated on the effect of competitive deposition of electroless Ni-W-P coatings on mild steel via a dualcomplexant plating bath composed of sodium citrate and lactic acid. Electroless Ni-W-P coatings were competitively deposited on mild steel by control of NaCit/Lac ratio and NiSO4 concentration in dual-complexant bath. The variations of the ratio and NiSO4 concentration affect the deposition rate of Ni, and correspondingly the co deposition of W. The bath at NaCit /Lac ratio of 1:1 and NiSO4 concentration of 30—40 g/L accelerates W, Ni deposition, but decelerates P deposition, so it incorporates more W (6.97— 7.02 wt.%) but less P (4.87—5.03 wt.%) in coating. Furthermore, the condition favors forming crystalline coating with homogeneous nodular morphology, and obtaining higher microhardness about 734—755 HV0.02 and adhesion around 36—38 N. The coating plated at different NaCit/Lac ratios and NiSO4 concentrations demonstrates various electrochemical and tribological behaviors. Detailedly, the smallest corrosion current density of the coating can be three magnitudes lower than the largest one, while the coefficient of friction of the coating varies in the range of 0.21—0.81, corresponding to the wear loss of 0.18—0.66 mg/min.

Hong Luo et al. [26] investigated on development of electroless Ni-P/nano-WC composite coatings and investigation on its properties. In this study, Ni-P-WC nanocomposite coating was prepared by electroless deposition methods, modifying the typical Ni-P coating through the addition of WC nano-particles. The morphology, structure, microhardness and corrosion resistance of the Ni-P-WC coating and conventional Ni-P coating were analyzed by using the optical stereoscopic microscopy (OSM), scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD), anodic polarization curve and electrochemical impedance spectroscopy (EIS). WC nano-particles and Ni-P deposited homogeneously on the Ni-P matrix, electroless deposited composite coating increased due to the existence of the nano-particles, and it will be improved after heat treatment. According to the results of corrosion testing in the 3.5 wt. % sodium chloride solution, the electroless Ni-P-WC coatings showed significantly improved corrosion resistance due to its special structure, compared to a conventional Ni-P electroless coating, even after 40 days immersion, it also exhibited good corrosion resistance ability.

Serin et al. [27] examined the effect of annealing temperature on hardness and wear resistance of electroless Ni–B–Mo coatings. We studied the formation of Ni–B–Mo coating on steel by electroless plating and evaluated their morphology, hardness, and wear resistance after applying heat treatments at different temperatures for 1 h. Ni–B–Mo coating exhibits higher hardness than the substrate material steel. The friction coefficient was at the lowest after heat treatment at 300°C; however, it increased with increase in temperature. Wear resistance was the lowest in coated sample, lower than the steel substrate. It reached the highest



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value after heat treatment at 300°C but did not change sharply up to 450°C; Serin observed a decrease in wear resistance in coating tempered at 550°C. Worn surface of the coatings showed deep grooves, demonstrating abrasive wear as the dominant wear mechanism. Tungsten detected on the surface of 550°C tempered coating after wear test emphasized an additional adhesive wear mechanism, which was further supported by the detected torn patch formation. Moreover, hardness of the Ni–B–Mo coating remained unchanged at higher temperature indicates higher thermal stability achieved through Mo addition. Therefore, Ni–B–Mo coatings have potential to be applied in precision mould, optical parts mould or bipolar plates, where thermal stability is essential. However, effect of heat treatment on corrosion is another important factor to be analyzed before considering the application of Ni–B–Mo coatings in the above mentioned areas.

Zahra Sharifalhoseini et al. [28] investigated on Ni-P coatings were deposited through electroless nickel plating in the presence and absence of ultrasound. The simultaneous synthesis of ZnO nanoparticle and its deposition under ultrasound were also carried out on the surface of Ni-P layer prepared by the classical method. The morphology of the surfaces and the chemical composition were determined by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), respectively. Electrochemical techniques were applied for the corrosion behavior studies. The Ni-P layer deposited by ultrasound showed a higher anticorrosive property than the layer deposited by the classical method. The ZnO nanoparticles deposited on the substrate surface of Ni-P layer significantly improved the corrosion resistance.

Fang et al. [29] examined to increase the corrosion resistance of stainless steel 316L in warm acidic solution, Ni–Cu–P coatings with high copper and phosphorus contents were deposited onto stainless steel 316L substrates via electroless plating. The structure of the film and its resistance to corrosion in a warm acidic environment were investigated using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction spectrometry (XRD), polarization curves, electrochemical impedance spectroscopy (EIS), and dipping corrosion tests, respectively. The results demonstrate that Ni–Cu–P coatings consist of two types of nodules, which are 19.98% Cu and 39.17% Cu (mass fraction) respectively. The corrosion resistance of the 316L steel substrate when subjected to a warm acidic solution is significantly incresed by the addition of the new type of the Ni–Cu–P coatings and those annealed at 673 K are found to corrode selectively, while pitting is observed to be the main corrosion mechanism of coatings annealed at 773 and 873 K.

Min Sun et al. [30] A graphene coating bonded on to stainless steel wire was fabricated and investigated as a solid-phase micro extraction fiber. The coating was characterized by scanning electron microscopy and energy- dispersive X-rays spectrometer. The graphene coating with rough and crinkled structure was about 1  $\mu$ m. These characteristics were helpful for promoting extraction. Using five n-alkanes (n-undecane, n-dodecane, n-tridecane, n-tetradecane and n-hexadecane), the fiber grain was evaluated indirect-immersion mode by coupling with gas chromatography(GC). Through optimizing extraction and desorption conditions, a sensitive SPME-GC analytical method was established. SPME-GC method provided wide linearity range (0.2–150  $\mu$ g/L) and low limits of determination (0.05–0.5  $\mu$ g/L). It was applied to analyze rain water and a soil sample, and analytes were quantified in the range of 0.85–1.96  $\mu$ g/L and 0.09–3.34  $\mu$ g/L, respectively. The use of TiO2 in coating improves Silanol resistance for basic conditions.

Yuxin Wang et al. [31] studied on the Ni-Co-TiO2 nano-composite coatings were electroplated on mild steel by adding transparent TiO2 sol (0-50 mL/L) into the Ni-Co plating solution. The microstructure, mechanical property and corrosion resistance of the composite coatings were systematically investigated. Wang found that after adding an optimum sol concentration into the plating bath, a good dispersion of TiO2 nanoparticles can be achieved in the Ni-Co coating matrix, resulting in a significant improvement in coating mechanical properties. Ni-Co, 12.5 mL/L TiO2 coating possessed the best microhardness, wear resistance and corrosion resistance. However, adding excessive quantities of sol (more than 12.5 mL/L) into the electrolyte caused nanoparticle agglomeration and created a porous structure, deteriorating the properties of coatings.

Xin Shu et al. [32] proposed that duplex Ni–P–ZrO2/Ni–P coating was electroless deposited onto stainless steel substrates. High phosphorus Ni–P coating was plated as the inner layer to improve the corrosion resistance and low phosphorus sol-enhanced Ni–P–ZrO2 coating was on the top to strengthen the mechanical property. The microstructure, mechanical property, corrosion resistance and hydrophobic property of coating were investigated systematically. The results show that the novel duplex coating possesses both excellent mechanical property and good corrosion resistance. The microhardness was further improved from 648 HV100 of Ni–P/Ni–P coating to 752 HV100 of Ni–P–ZrO2/ Ni–P coating, while the hardness of stainless steel is below 200 HV100. The duplex coating has very good corrosion resistance due to the different corrosion mechanism compared with the single Ni–P coating. This duplex coating also shows hydrophobic property.

R. A. Shakoor et al. [33] reprted that Electroless Ni-B coatings have attractive properties such as uniform thickness, decent corrosion resistance, good wear and abrasion resistance, good solderability, high hardness, low coefficient of friction, high



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reflectivity, low resistivity and good magnetic properties. Furthermore, their inherent properties can be further improved either by the addition of alloying elements, second phase particles or by the process of heat treatment and nitriding which make them suitable for surface modification of a large number of materials like steel, aluminum, copper, magnesium alloys, plastics etc. Owing to their promising mechanical properties (hardness, wear), magnetic and decent electrochemical properties (anticorrosion), the electroless Ni-B coatings are considered attractive for food, petroleum, petrochemical, chemical, plastics injection, optics, aerospace, fire arms, automotive and electronics industries.

Kang Huang et al. [34] studied the controlling formation, establishment, and proliferation of microbial biofilms on surfaces is critical for ensuring public safety. Herein, Kang reported on the synthesis of antimicrobial nanoparticles and their co-deposition along with fluorinated nanoparticles during electroless nickel plating of stainless steel. Plating bath composition is optimized to sufficiently low surface energy to absorbs fouling and microbial adhesion as well as exert significant (>99.99% reduction) antimicrobial activity against Listeria monocytogenes. The resulting coating presents hybrid antifouling and antimicrobial characteristics, can be applied onto stainless steel, and do not rely on leaching or migration of the antimicrobial nanoparticles to be effective. The resulting coatings can support reducing public health issues related to microbial cross contaminations in areas such as food processing, hospitals, and water purification.

Ulas Matik et al. [35] studied the structural and wear properties of Ni-P-Si3N4 plated and heat-treated electroless Ni-P alloy and Ni-P-Si3N4 composite coatings on iron based powder metal (PM) compacts. The coatings were prepared using hypophosphite reduced alkaline electroless nickel bath. The synthesized submicron α-Si3N4 particles (10 g/l) were mixed into the Ni-P bath to develop Ni-P-Si3N4 composite coatings on the PM compacts. The coatings were heat-treated at 300, 400 and 500 °C, for 1 h and the effect of heat treatment on the coating performance was studied. Chemical composition of the coatings was analyzed by EDX. The phases and morphology of Si3N4 particles and coatings were examined by XRD and SEM. Phase transformation behavior was studied by differential scanning calorimetry (DSC). The wear properties were studied using pin-on-disc tribometer under unlubricated sliding conditions under a normal load of 40 N, sliding speed of 1 ms-1 and sliding distance of 1000 m. The results show that the incorporation of submicron Si3N4 particles in the deposit significantly has been found to improve the hardness and wear resistance of composite coatings. The hardness and wear resistance of Ni-P and Ni-P- Si3N4 coatings heat-treated at 400°C had the maximum hardness and wear resistance.

A.P.I. Popoola et al. [36] reported that sliding wear analysis shows considerable increase in the wear resistance of the mild steel substrate, attributed to the electroless deposition. The microstructure incorporated after Sn addition was the major reason for the improvement of corrosion and wear resistance of the mild steel.

M. Ghaderi et al. [37] examined the Ni–B–Al2O3 composite was successfully coated on the surface of Ck45 steel by elecroless method. X-Ray diffraction analysis (XRD) and scanning electron microscopy (SEM) were analyzed in order to investigate and identify the coating properties. Wear behavior of the coating was studied by the pinon- disk test. Corrosion behavior of the Ni–B and Ni–B– Al2O3 coatings was investigated by using Tafel polarization diagrams in the 3.5% NaCl solution at room temperature. The obtained data demonstrate that the addition of Al2O3 nanoparticles to the coating has resulted in improving the tribological behavior of the coating due to the presence of the composite nanoparticles. Also, the results of electrochemical testing show that corrosion resistance of the electroless Ni–B coating with Al2O3 nanoparticles has increased.

A. Sadeghzadeh-Attar et al. [38] studied that SiO2 nanoparticles were successfully applied to electroless Ni-P composite coating via addition of transparent sol to the conventional electroless solution. This novel method of introducing nanoparticles by adding SiO2 st-sol solution could effectively avoid particle agglomeration. The mechanism of SiO2 nanoparticle formation was also studied in detail in terms of the hydrolysis and condensation. The SEM images showed the presence of SiO¬2 nanoparticles, in which the absence of any agglomerates from the microstructure of coating confirmed the uniform dispersion of SiO2 nanoparticles throughout the surface of the Ni-P matrix. The hardness and tribological properties of coatings are improved in the presence of SiO2 nanoparticles and hard crystalline precipitates of Ni and Ni3P, which prevent grain growth and mobility of matrix dislocations. A very good correlation between the surface morphology, chemical composition, microhardness, and tribological behavior of coatings was found in this investigation. The wear mechanism was also studied and found the dominant mechanism for wear of coatings was abrasive wear.

M. Czagánya et al. [39] examined the effect of the P-content in the Ni-P coatings was experimentally studied on the thickness of the coating, on its nano-micro-structure and on its micro-hardness. The steel samples were nano-crystalline (mostly amorphous according to XRD) and their micro-hardness was found to decrease with increasing the P-content. Upon annealing at 400°C a new Ni3P phase was formed and the nano-crystalline Ni-rich grains coarsened to micro-grains. In this annealed state the micro-hardness was found to increase with increasing the P-content results. The



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as-received Ni-P coating contains almost pure Ni nano-grains surrounded by segregated P atoms; as the grain grows, its surface is covered more and more by the P atoms. If the grain is fully covered by the P atoms, further grain growth is restricted and the coating can grow further only due to nucleation of a new grain. Hence, the size of the grains was found inversely proportional to the Pcontent of the Ni-P alloy. The need for a larger number of nucleation events with decreasing grain size explains why the coating has a smaller thickness for smaller grain size, i.e. higher P-content. The inverse Hall-Petch rule was found for the grain size dependence of micro-hardness of the as deposited samples due to the grain boundary sliding of relatively hard Ni-rich nano-crystals along the soft P atoms (higher P-content lead to lower micro-hardness through smaller grain size). After annealing the micro-hardness was found to increase with the volumetric phase fraction of the harder Ni3P phase within a relatively soft Ni matrix, i.e. it was found to increase with the P-content of the Ni-P coating. The extrapolated value for the micro-hardness of the Ni3P phase is found about 757  $\pm 20$  HV.01.

Shalini Mohanty et al. [40] The purpose of this study is to deposit a layer of Ni–P and Ni– Al2O3– WS2 composite coating by varying the concentration of Al2O3 and WS2 nano-powders in the Ni–P plating bath. A comparative study has been carried out by adding nano-powders into the Ni–P bath and without the use of powders. The incorporation of powder in the bath yields certain desirous properties to the substrate surface that finds industrial applications. The mechanical properties and surface characteristics are enhanced by varying the powder concentration in the electroless Ni–P plating bath. To analyze the elements present in the coated layers and study the surface morphology, FESEM along with EDX has been used. In addition to that, the intermetallic compounds have been identified by using XRD. The micro-hardness of the coated samples without addition of powder is seen to be improved (789.02 HV) when compared to that of the substrate (76.68 HV). However, adding the nano-powders in the bath shows fluctuation in micro-hardness ranging from 476.37 to 1286.58 HV. It is probably due to the dispersion hardening effect of both the powders into the substrate material. Although the maximum coating thickness is obtained with 3 g/L Al2O3 and 1.4 g/L WS2 powder concentration, uniform thickness is seen at 4 g/L Al2O3.



Fig 2.3 shows the variation of Hardness VS WS2 with varying aluminum oxide concentration.

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