# Synthesis of electroless Ni-P/Ni-P-W nanocomposite platings and sustainable tribological characteristics

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***Abstract***

*The tungsten (W) is an exceptionally strong refractory metal of group VIB of periodic table and has the highest melting point, the lowest coefficient of thermal expansion with high neutron capturing capability and can remove sulphur from crude oil. Therefore, in this investigation, the synthesized sodium-tungstate nanoparticles ($Na_2WO_4$, 0.5±0.01gpl in amount and 40-150 nanometer in size, Zirox technologies) were unsteadily dispersed into an acidic electroless Ni-P bath (pH=05.6) for Ni-P-W nanocomposite deposits. Consequently, as an outcome of the experimental work the Ni-P/Ni-P-W nanocomposite deposits with thicknesses ranging from 03.2 to 14.7-micron meters were produced on the mild steel (MS; AISI1040 grade) substrates. The SEM, EDAX and XRD instrumental methods were also used to appraise the surface morphology, elemental contents and phases of deposited Ni-P/Ni-P-W nanocomposite deposits. The domino effect of these studies illustrate that the Ni-P-W nanocomposite deposits have small whitish colour homogeneous husky globules of tungsten (W) nanoparticles into the electroless Ni-P matrix.*

*Furthermore, the daintily created Ni-P/Ni-P-W deposits were tested for wear losses and microhardness values using a wear tester (model TR-20LE-CHM-400; Ducom) and a Vicker microhardness tester (VMHT-MOT). The wear and microhardness tests were carried out in accordance with the ASTM G99 standard with varying loads and deviations over an invariable 250.0-meter distance (wear test). Consequently, the inclusion of tungsten (W) nanoparticles into the acidic electroless Ni-P matrix had a noteworthy impact on wear and microhardness resistance and can be used as an alternate of hard chrome-platings. The results can be arranged into the following manner: Ni-P-W (heated at 400°C) > Ni-P-W (heated at 600°C) > Ni-P-W (heated at 200°C) > Ni-P (heated at 400°C) > Ni-P (as-deposited) > MS.*

**Keywords:** Electroless, nanocoatings, Ni-P-W, microhardness, wear resistance.

## Introduction

The surface coating industry is facing large challenges to replace chrome plating as the chromium (Cr) causes skin irritation, ulceration, damage to liver, kidneys, nerve tissue and cancer etc. Therefore, chromium reduction has been a significant focus of companies, industries, academia and legislation etc.[1,38] The electroless nickel (EN) has been used to substitute chrome into numerous decorative as well as functional applications, such as for corrosion and wear resistance etc.[2] In applications requiring hardness and wear resistance, the composite electroless nickel coatings have been even more efficacious in not only replacing chrome but essentially surpassing the enactment of hard chrome plating[33,35]. The composite electroless nickel coatings are regenerative because of the uniform manner with which the particles are dispersed throughout the entire plated layer as observed in the cross sectional figure 1[3,16,17].

Certain performance benefits have been discovered when a composite coating is generated simultaneously using two distinct particle sizes and incorporation can be from nanometers to around 10 microns in size. It is hypothesized that the smaller particles fill the spaces between the larger particles. This also further increases the percent by volume of the particulate matter and further reduces the amount of nickel used[36]. Composite EN coatings have also all the inherent features of electroless-nickel as well as the properties of whatever particles are selected such as hardness, wear resistance, lubricity, heat transfer, light absorption etc. For this reason, composite EN coatings are better than chrome or any electrolytic or spray processes for non-line-of-sight applications[4,42,55].

The wear and corrosion phenomena are incredibly very severe and mainly accountable for life of machineries along with process equipment into typically chemicals and petrochemicals and other industries contaminating the environment[14,23,44,48]. The several other in-plant and electrochemical corrosion studies performed on peroxides, chlorides, peracids and sulphide environments concluded that the higher grade austenitic and duplex stainless steels are not suitable materials into these environments[2,19,47-54]. The harsh outcomes due to corrosion and wear phenomena can be minimized or stopped by diverse protective techniques and methods such as employment of higher grade materials, proper protective surface coatings (electroplating and electroless platings, CVD, PLD etc.) over the metals or alloys that are most prone to corrosion and wear[17,22,24,26].

More importantly among the available surface protective methods, the electrodeposited (ED) and electroless (EL) deposition methods have been suggested to be the best promising alternatives methods[1,8,9,11,16]. It has been reported in one study that the copperu-nickeli-chromium (Cu-Ni-Cr)

deposition, on the magnesium (Mg) and other alloys has shown fine corrosion resistance into mildly corrosive conditions[29]. Moreover, several authors alluded examples of chromium (Cr) gratis namely electrodeposited and electroless Ni, Cu, Ni-P, Ni-B coatings etc. These coatings more specifically have been applied successfully into chemicals, automobile, paint, food, paper, medical, nuclear, computer as well as electronics industries for corrosion and tribological resistance applications[5,9,13,33,35,38].

Some review papers also have discussed proposed depositing of diffusion types such as chromates, phosphates, fluorides, stannates, rare-earths elements etc. by electrodeposit along with electroless procedures and revealed that the electroless deposition process does not suffer the disadvantages that are associated with the electrodeposit process. Additionally, in the electroless deposition method (Figure 1), there is the possibility of codeposition of second phase micro and nanoparticles for improving microhardness, abrasive and corrosion resistance properties and following equations can be written for electroless nickel phosphorous (ENP) deposition on a substrate as[1-3,14-18,21,22,25,45,52,55].

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + 2H^+ + 2e^- \quad (1)$$

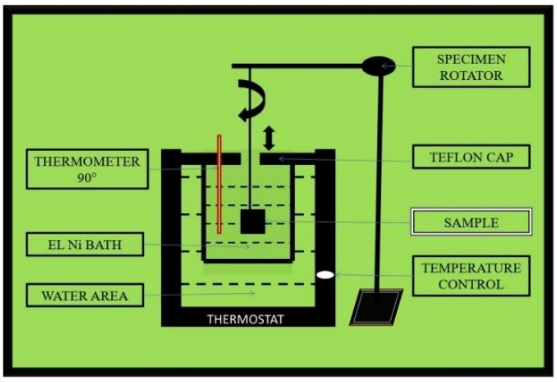
$$Ni^{+2} + 2e^- \rightarrow Ni \quad (2)$$

$$2H^+ + 2e^- \rightarrow H_2 \quad (3)$$

$$H_2PO_2^- + 2H^+ + e^- \rightarrow P + 2H_2O \quad (4)$$

The literature also cites some corrosion and wear resistance related studies on electroless Ni-P coatings[8,9,21,46]. The inclusions of another phase micro/nanoparticles (X) into Ni-P electroless medium, are recognized as electroless (Ni-P-X) nano-dispersion deposits and these deposits can be very useful into those special circumstances where one necessitates an amalgamation of very good tribological along with fine corrosion resistance properties[1,3,14-18,25,45]. The (Ni-P-X) nano-dispersed deposits have two diverse groups of nanoparticles such as spongy nanoparticles (PTFE, Graphite, $MoS_2$ etc.) for lower friction-coefficient and hard nanoparticles ($Al_2O_3$, SiC, $TiO_2$, $B_4C$, TaC, WC, $ZrO_2$ etc.) for imparting hardness and abrasion resistance to electroless nickel-phosphorus (Ni-P) coatings[4,7,10,15,20,27-41,51,57,58]. The electroless Ni-P-X platings are anticipated to have intermediary values of microhardnesses, tremendous abrasion, wear and corrosion resistances with low friction coefficients.

Furthermore, these hard platings under mildly corrosive conditions can be used for fabricating pistons rings of engines, mould, electrodes materials, automobile's part and common wear constituents into the manner and other industries. In recent research activities, the electroless Ni-P-W, Ni-P-$Si_3N_4$, Ni-P-CNF, Ni-P-ZnO, Ni-P-$WO_3$ and Ni-P-$TiO_2$ etc. coatings have demonstrated superior microhardness, corrosion and wear resistances ended Ni-P alloys and can be used in many industries.[6,14,34,43,44,48-51,56].

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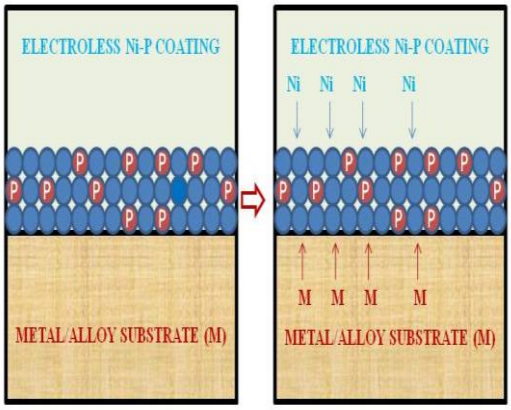



**(b)**

**Figure 1: (a) Schematic Photograph of deposition of Ni-P-W nanocomposite coatings (b) Atomic layer deposition mechanism process**

The microhardness along with corrosion resistance, electrochemical impedance (EIS) and friction behavior etc. for the electroless Ni-P-W platings is carried out in many studies[4,7,29-31,56].

Additionally, worldwide, more significant efforts are too being made for inclusion of tungsten (W) nanoparticles into acidic electroless Ni-P coatings because of its inimitable chattels such as a transition element, good thermal stability, removing sulphur from crude oil and very high melting point (3422ºC), very good corrosion and wear resistance properties.

Therefore, in the present experimental work, the tungsten nanoparticles are inserted into electroless Ni-P matrix for examining the trioblogical behavior of acidic electroless Ni-P-W nanocomposite platings in as-plated and heat-treated conditions which will be directly beneficial for many industries and indirectly for environment[4,7,56].

## Material and Methods

The acidic Ni-P/Ni-P-W platings were deposited on (MS, AISI1040) substrates, through an electroless method. The chemicals compositions of mild steel (MS) base substrates are defined as carbon (C=00.18%), manganese (Mn=01.66%), silicone (Si=00.04%) and iron (Fe=Balanced). All coupons before electroless deposition were polished through silicon carbide (SiC) emery papers having grade from 50 upto 1200 grit and later on at once cleaned ultrasonically as well as with acetone ($CH_3COCH_3$)[2,47,53,54]. The ultraclean substrates were first sensitized by 1.0% $SnCl_2$ (for 120 seconds) and after it activated through a 0.05% $PdCl_2$ solutions (for 30 seconds). These both reagents ($SnCl_2$ and $PdCl_2$) as catalytic sites, aimed at nucleation throughout electroless deposition (ED) process.

During electroless deposition (ED) process, the sodium tungstate ($Na_2WO_4$) nanoparticles (amount; 0.5±0.01 gpl, size; 40-150 nm) were separately added to the electroless Ni-P bath. A miniature Teflon-plated cylindrical magnet was applied to stirring Ni-P electroless bath mixture continuously. Previous to the Ni-P deposition, pH of electroless bath was adjusted and kept stationary at 05.6 and temperature variation in between $83^0$C to $87^0$C. The total deposition time was selected for only two hours (30 minutes for Ni-P coating and 90 minutes for tungsten nanoparticles) and suspension of the co-deposited nanoparticles (tungsten) was kept homogeneous by unremitting stirring.

After plating process is over, the as-plated specimens were first air dried at room temperature and then heated up to required temperature. For heat treatments processes, the as-plated specimens were retained at $200^0$C, $400^0$C and $600^0$C for one-hour duration in tube furnace (ESE, Howarah, RT-$1450^0$C) with argon (Ar) gas atmosphere and rate of heating was kept $10^0$C per minute and at that moment were ventilated (annealed) into a furnace to the room temperature and later on used for more experimental investigations (Table 1).

The as-plated and heated specimens were characterized by Scanning electron microscopy combined with energy dispersive X-ray analysis (FEI; QUANTA FEG 200 model) instrument and X-ray diffraction (XRD; Bruker AXS D8 model) instrument for microstructural, elemental composition, elemental concentration and recognizing the phases respectively.

**Table 1**
**Chemical compositions and established electroless bath parameters**

| S.N. | Salts/chemicals formulae | Gramm (gm) of the amount dissolved in 100 milliliters of de-ionized water | Functions of used chemicals |
|---|---|---|---|
| 1 | $NiSO_4.6H_2O$ (make of sigma-aldrich) | 04.18 gm | Serve as a source of $Ni^{2+}$ ions. |
| 2 | $C_6H_5Na_3O_7.2H_2O$ (merck) | 04.70 gm | Function as a complexing negotiator to prevent the release of $Ni^{2+}$ ions without control. |
| 3 | Sodium Acetate | 02.54 gm each | A buffer that is acidic to maintain a pH of 05.6 |
| 4 | NaOH/ $CH_3COOH$ 10 % solution | add drop wise | To keep the solution's pH at 05.6 |
| 5 | $NaH_2PO_2.H_2O$ (loba chemicals) | 02.47 gm | Reduces and provides electrons to $Ni^{2+}$ ions |
| 6 | $Na_2WO_4$ nanoparticles (40–150 nm, Zirox make) | 00.05 gm | Reinforcement in electroless Ni-P matrix |
| 7 | Operating parameters for a bath | - | The 83-87°C temperature range; and continuous stirring is necessary |
| 8 | Annealing temperatures | 200 to $600^0$C for one hour in a pure 99% argon environment in steps of $200^0$C | To understand impact of heat performance on the coated specimens |

The middling grains sizes of all plated specimens were estimated from penetrating Ni (111) peaks through using Scherer equation[12,46]. The microhardness measurements on all the plated specimens were carried out by using Vickers microhardness tester (VMHT-MOT), after applying a load of 25.0 gmf. The average of five experimental thicknesses on every-one two diverse specimens was considered. The measured microhardness honors have been characterized on $HV_{25}$ scales of microhardness. The wear tests were directed through using pin-on-disc wear tester (TR-20LE-CHM-400) and having a diamond (C) coated pin of 0.8 cm in diameter and 3.0 cm in length. For the wear tests, a load of magnitude 05.0N, 15.0N and 25.0N was pragmatic on the platted pin. Wear resistance of all coated specimens was predicted afterward a whole sliding distance of 250.0 meter and through measuring a weight loss in coated specimens.

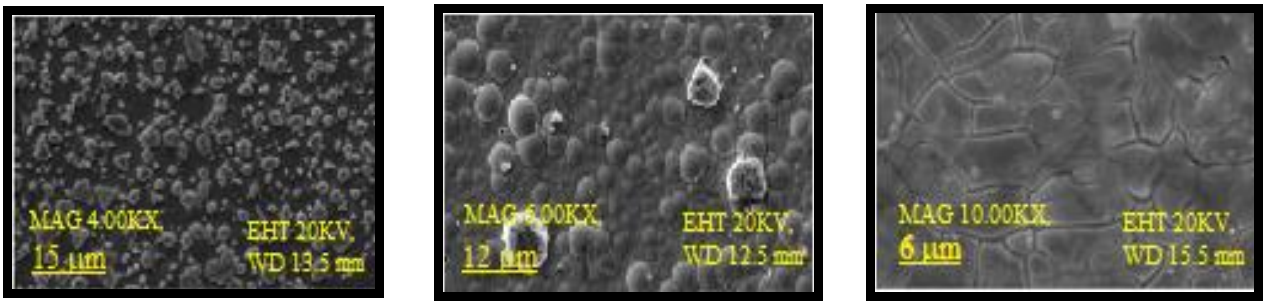
Moreover, dilapidated surfaces of coated specimens were analyzed through a Scanning electron microscope system. Furthermore, during wear tests, the frictional force was illustrious at customary time intermission for each coated specimen conforming to a specified pragmatic load. The middling of these calculated values (for deposits at a specified functional load) was noted to find approximately the roughness coefficient through dividing this middling charge of friction-force through a specified functional load.

## Results and Discussion

**Characterization of Coating:** The Scanning electron microscopic (SEM) images of electroless Ni-P/Ni-P-W nanocomposites as-plated and heated (200, 400 and $600^0$C) platings are illustrated in figure 2. The SEM micrographs of as-plated Ni-P-W nanocomposite coatings specified the existence of numerous globules on the mild-steel (MS) substrate surfaces with the inclusion of apparently another phase tungsten (W) nanoparticles. These globules were identified as tungsten (W) nanoparticles and are also ostensible from the EDAX data's values (Table 2, Figure 3).

Moreover, through SEM micrographs of Ni-P-W heated platings, it is noticeable that blobs sizes in these coatings prolong to nurture. After heating treatment at $400^0$C, the blobs into platings have developed to magnitude that they come into sight to have coalesced into each other. In Ni-P-W heated specimens from EDAX study, it is observed that platings components amount of nickel (Ni), phosphorus (P), tungsten (W) decreases whereas in case of iron (Fe) it increases and could be characteristics of dissemination of these plating elements towards inter-face of Ni-P-W deposition and the mild-steel (MS) substrate.

The X-ray diffractograms of as-deposited and heated platings are evident for simulation of a widespread peak of nickel (Ni) at $43.5^0$ and some other weak peaks corresponding to tungsten (W) at ($54.7^0$) and of iron (Fe) as a fragment of mild-steel (MS) coupon at $64.7^0$ and $81.2^0$ (Figure 4). The extensive peak is a pin-pointing of amorphous character of deposited platings. The X-ray diffractograms of heated platings at $200^0$C do not reveal any noteworthy change as compared to as-plated platings.




**(a)** **(b)** **(c)**

**Figure 2: SEM images of (a) Ni-P-W as-plated coating (b) Ni-P-W heated at $400^0$C coating (c) Ni-P-W heated at $600^0$C coating**

**Table 2**
**EDAX analyses of Ni-P-W nanocomposite coatings**

| Elements | Weight % | | | |
|---|---|---|---|---|
| | Ni-P-W (as-coated) | Ni-P-W (heated 200 $^0$C) | Ni-P-W (heated 400 $^0$C) | Ni-P-W (heated 600 $^0$C) |
| Ni K | 83.23 | 82.81 | 81.12 | 79.59 |
| P K | 09.72 | 09.02 | 08.63 | 08.18 |
| Fe K | 01.31 | 02.47 | 03.97 | 04.16 |
| W K | 03.42 | 03.21 | 02.75 | 02.63 |
| O K | 02.32 | 02.49 | 03.53 | 05.44 |
| Total % | 100.00 | 100.00 | 100.00 | 100.00 |

Although, heated platings at $400^0$C and $600^0$C expose some extra peaks and less broad as, it is because of formation of the inter-metallics of Ni-P alloys (bct) precipitates and W-P alloys and mountains of Ni $43.9^0$, $51.3^0$, $75.5^0$, $Ni_3P$ $42.1^0$, $46.5^0$,$43.9^0$, $Ni_7P_3$ $42.4^0$, $46.9^0$, $Ni_2P$ $46.7^0$, $45.3^0$, $Ni_5P_4$ $30.5^0$, $36.5^0$, $50.5^0$, $Ni_{12}P_5$ $50.1^0$,$47.5^0$, $37.3^0$ and W-P $33.1^0$,$47.5^0$,$55.5^0$ observed as key components (JCPDS 000031044)[7,12,29-32,35-39,56]. The grains size calculated by Scherer formula for Ni-P-W platings shows that as the temperature increases, the grains size too increases and it is varied from 01.7nm to 07.3nm for $200^0$C heated specimen,07.5nm to15.1nm for $400^0$C heated specimen and finally 20.2 nm to 37.3 nm for $600^0$C heated specimen[7,12,29-39,56].

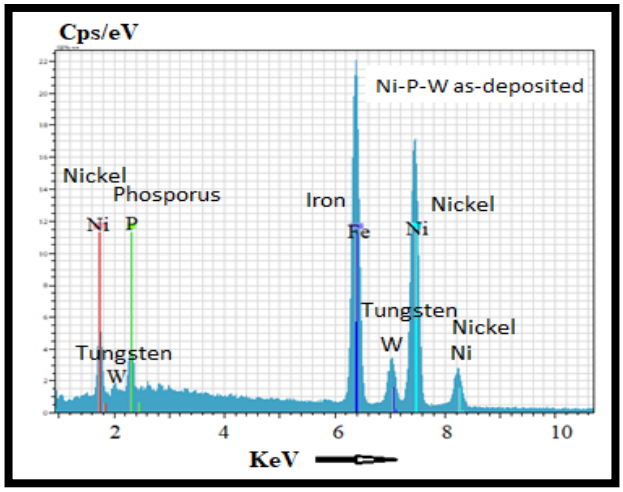
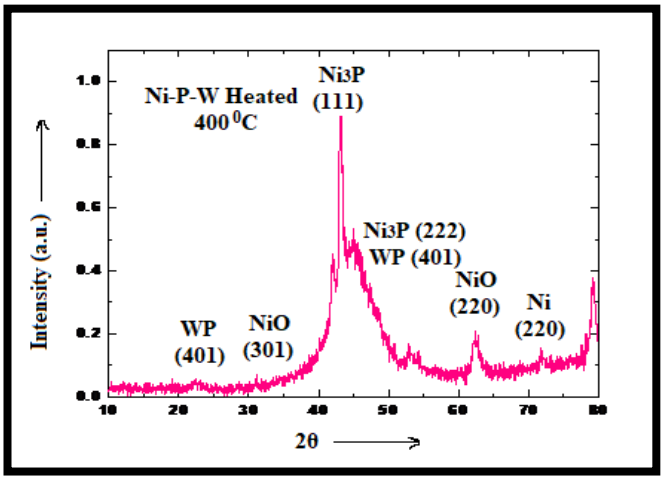



**Figure 3: EDAX photographs of Ni-P-W as-deposited plating**

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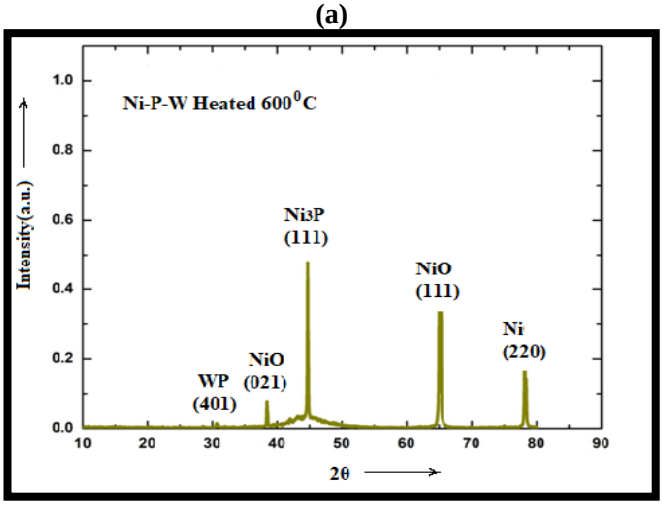



**(b)**

**Fig. 4: (a) XRD photograph of Ni-P-W heated platings heated at 400°C (b) XRD photograph of Ni-P-W heated platings heated at 600°C**

The microhardness values at $HV_{25}$ scale and micrographs of Ni-P-W as-plated and heated coatings are described and illustrate generally that as temperature increases the microhardness tenets of Ni-P/W deposits increase (Table 3).

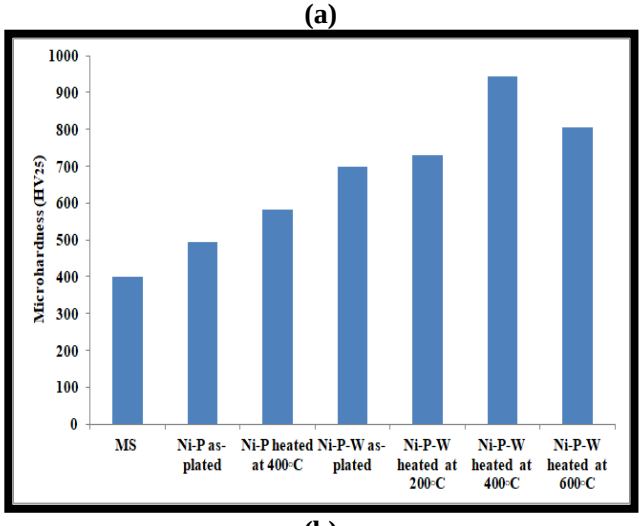
The highest value of microhardness is observed for specimen heated at $400^0$C and this might be attributed to rapid establishment of inter-metallics of Ni-P, W-P structures and is apparent through the X-ray diffraction study[7,12,35]. Exceptionally, decline into microhardness for heated coatings at $600^0$C looks like owing to abrading of grains (Figure 5).

Moreover, as abrading of grains phenomenon occur, the decline into grains margins area takes place. The wear losses calculated through pin-on-disc test method for mild steel (MS) and further Ni-P/Ni-P-W electroless nanocomposite depositions, establish considerable miscellaneous wedged between as-deposited along with heated Ni-P-W platings for 05.0N, 15.0N and 25.0N loads (Table 4).

**Table 3**
**Microhardness value of diverse tested coupons**

| Nomenclature of tested coupons | Microhardness values of coupons at $HV_{25}$ gauge at different temperatures |
|---|---|
| MS | 398.71 |
| Ni-P as-plated | 493.19 |
| Ni-P heated at 400˚C | 580.52 |
| Ni-P-W as-plated | 697.89 |
| Ni-P-W heated at 200˚C | 731.08 |
| Ni-P-W heated at 400˚C | 944.48 |
| Ni-P-W heated at 600˚C | 806.41 |


**(a)**

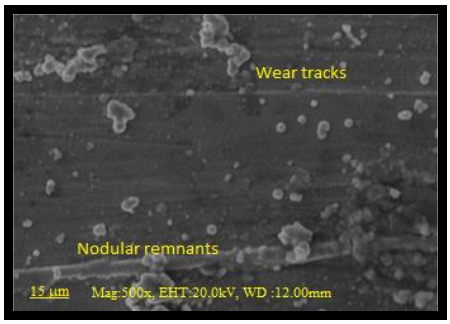



**(b)**

**Figure 5: (a) Microhardness indentation of a tested Ni-P-W heated coupon at 600˚C (b) Microhardness values of various tested coupons**

**Table 4**
**Wear losses of diverse tested coupons**

| Nomenclature of tested coupons | Weight loss into (mg) of specimens at applied loads along with pin sliding speed 0.20 m/s and distance 250 meter | | |
|---|---|---|---|
| | **05.0N** | **15.0N** | **25.0N** |
| MS | 11.91 | 17.38 | 21.18 |
| Ni-P as-plated | 06.42 | 07.89 | 08.28 |
| Ni-P heated at 400˚C | 05.39 | 06.49 | 07.86 |
| Ni-P-W as-plated | 03.97 | 04.68 | 05.38 |
| Ni-P-W heated at 200˚C | 03.83 | 04.27 | 04.97 |
| Ni-P-W heated at 400˚C | 02.20 | 02.54 | 03.18 |
| Ni-P-W heated at 600˚C | 02.11 | 03.28 | 03.77 |

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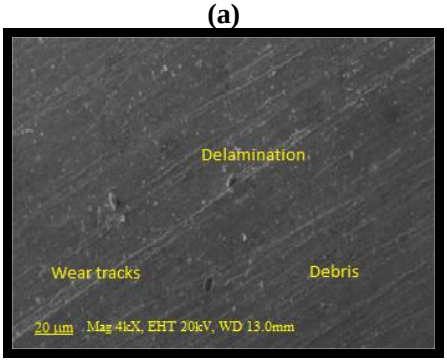


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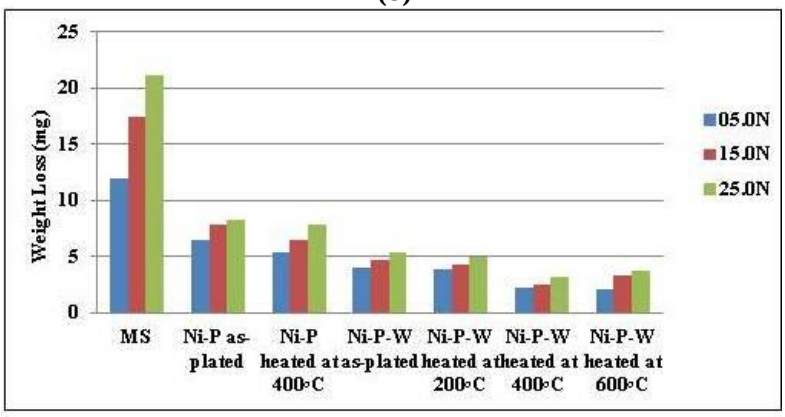


**(c)**

**Figure 6: (a) and (b) SEM photomicrographs of Ni-P-W platings subsequently wear tests at 05.0N load and 400˚C temperature and 25.0N load and 600˚C temperature respectively and (c) Weight loss in wear tests of various tested coupons**

Further, the wear losses are low for Ni-P-W heated nanocomposite depositions as associated to the Ni-P-W as-plated and mild-steel (MS) specimens and are endorsed to augmented microhardness of these depositions. Continually since boost into microhardness values is observed finest at $400^0$C, the wear thrashing is besides hardnosed deepest for this depositing (Figure 6)[29-32,35].

The coarseness coefficient, for Ni-P-W as-plated plating is 0.36 and this value is boosted to 0.57 for heated plating at

$400^0$C temperature for 25N load. This enhancement into friction coefficient might be accredited to increase into microhardness of this plating. From wear tests, the dilapidated surfaces of Ni-P-W as-deposited along with heated deposits ensure missing scratches upon surfaces of Ni-P-W heated platings. Moreover, in Ni-P-W heated coupons, the inter-metallics of Ni-P, Ni-W and W-P formed are unbendable and feeble into character. Submissive adhesive nature of wear surface is also accountable to exhibit a reduced wear loss amount[35-39,56].

## Conclusion

This investigation describes synthesis of electrolessly deposited Ni-P/Ni-P-W nanocomposite platings and heating effects on microhardness and wear losses. It can provide a very good alternate of hard chrome platings. The Ni-P/Ni-P-W as-deposited depositings demonstrate an amorphous atmosphere and on heating up to $600^0$C into step ladder of $200^0$C, these coatings show decline into amorphous character and crystallizations of inter-metallics of Ni-P/W-P alloys. Moreover, above $400^0$C temperature, the grains evolution starting is pragmatic and these transformations have been recommended to cause boost into microhardness, friction coefficient as well as wear resistance.

Moreover, the grains evolution into case of $600^0$C treatment temperature results in lowering microhardness and is recognized as formation of greater grains boundary areas as equated to an amorphous nature, establishments of bimetallic cells between Ni matrix and inter-metallics, reduction into quantity of phosphorous and un-bonding between mild steels substrates and coatings due to their differential thermal enlargement. The investigated specimens revealed microhardness and wear resistance into following directives as: Ni-P-W heated at $400^0$C > Ni-P-W heated at $600^0$C > Ni-P-W heated at $200^0$C > Ni-P-W as-plated > Ni-P heated at $400^0$C > Ni-P as-plated > MS.

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