



## A Comparison between the Effect of Heat Treatment and Plasma on the Wear Activities and Corrosion Resistance of Ni-B Electroless Coating with AISI 4340 Steel

<sup>1</sup>Abdul Raheem K. Abid Ali\*, <sup>2</sup>Alaa M. Wais

<sup>1</sup>Department of Metallurgy Engineering, College of Materials Engineering, University of Babylon – Iraq

<sup>2</sup>Biomedical Engineering Department, Al-Mustaqbal University College – Iraq

### Article information

#### Article history:

Received: May, 18, 2022

Accepted: July, 31, 2022

Available online: October, 20, 2022

#### Keywords:

Electroless coating,

Ni-B,

Heat treatment

#### \*Corresponding Author:

Full Name: Abdul Raheem K. Abid Ali

[alaawaies05@gmail.com](mailto:alaawaies05@gmail.com)

#### DOI:

<https://doi.org/10.53523/ijoirVol9I2ID167>

### Abstract

The study the hardness, wear corrosion resistance of as-coating, heat treatment and plasma-nitriding of Ni- B electroless coatings was deposit on 4340 steel. After the procees of coating, samples were plasma-nitriding in percentage hydrogen/nitrogen ratio (50%), in the temperature 400°C, at 4 hours were compared with heat treatment. characterization by of FESEM, XRD, microhardness, Corrosion Resistance and surface roughness measurements. Microhardness was show highest hardness of 1050 HV was obtained for Ni-B plasma-nitriding formula whereas the greatest hardness of 800 HV was become for sample of Ni-B heat treated. Ni-B coating is used in the enhancement of properties of the corrosion of AISI 4340 steel is due to the thickness of electroless deposition, which is uniform, and the cold of Ni-B coating to act as an active barrier between corrosion media and metal, it should be noted that the value of E<sub>corr</sub> is less negative and the rate of corrosion and I<sub>corr</sub> is lower on the plated sample than on the uncoated sample. The rate of the wear for samples is decrement by plasma-nitriding with deposition time at (4h) and the temperature 400°C. Rate of wear for the materials depends not just into condition of wear test, but also with several properties of material and features as topography, hardness and friction coefficient.

### 1. Introduction

Plating technique by using of aqueous solutions obtain a great consideration due to the aims like easy of the coating technique, large rate of deposition, lower cost, uniform layer of plating [1-4]. The technique has feature like larger hardness, thickness, resistance wear, good solderability, good resistance of corrosion, deposit of amorphous or microcrystalline, lower coefficient of friction, lower resistivity and excellent magnetic properties [5-10]. Technique of Ni-B the electroless coating is obtaining to good the surface properties for larger types of substrates [11-12]. Commonly, technique of Ni-B electroless coating is thought to be better when compared with Ni-P coating also it more attractive in many industries [13-16]. Improved of mechanical properties, wear resistance and resistance of corrosion, and coatings of nanocomposite have great deal of attention. Nanocomposite coating has another appropriate identified solid lubricant like MoS<sub>2</sub> [17], PTFE [18], Cu-CNT [19], and Ni-P-CNT [20]. larger

tensile strength for carbon nanotubes with elastic modulus, result it used in ceramic and metallic composites, In plasma nitriding, the nitrogen is presented to the surface of the substrate when being diffused with the metal. The energy of the large electrical voltage is formed on the plasma when atoms of nitrogen are accelerated to impinge on the substrate and Figure (1) Show diagram planner of plasma nitriding system [21-27]. Studying effects behaviour for Ni-B electroless coatings with Plasma nitriding and heat treatment coating on microhardness and roughness, resistance of corrosion and microstructure of 4340 steel.

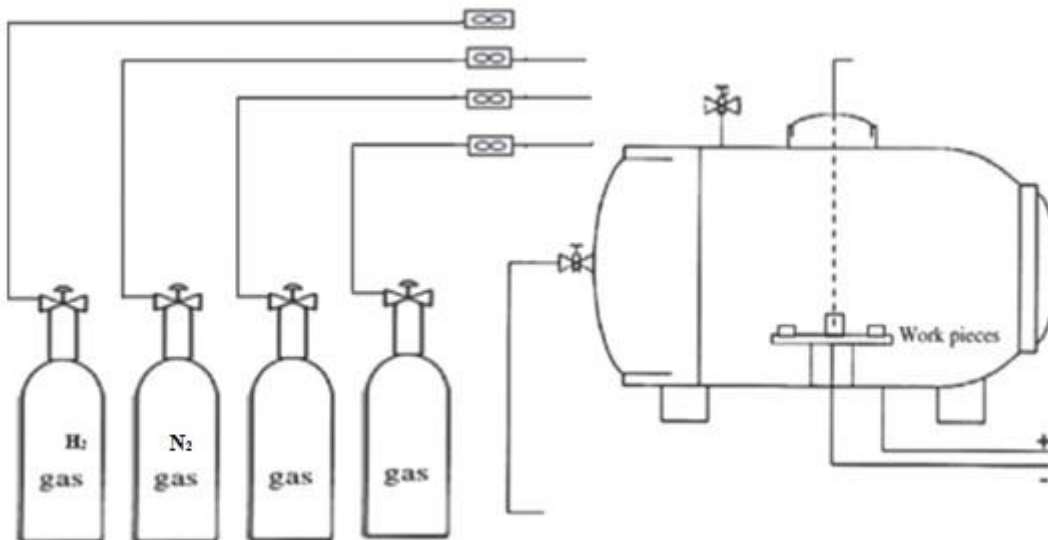


Figure (1). Show diagram planner for the plasma nitriding system

## 2. Experimental Procedure

### 2.1. Preparation of Substrate

The substrate metal used is 4340 steel. Samples (diameter (20 mm) × height (10 mm)) were used as the base metal. The analysis of chemical for 4340 steel is shown Table (1).

Table (1). Chemical composition of 4340 Steel.

Element	C	Si	S	Mn	P	Cr	Ni	Mo	Fe
W%	0.36	0.29	0.01	0.67	0.01	0.81	1.3	0.15	Bal.

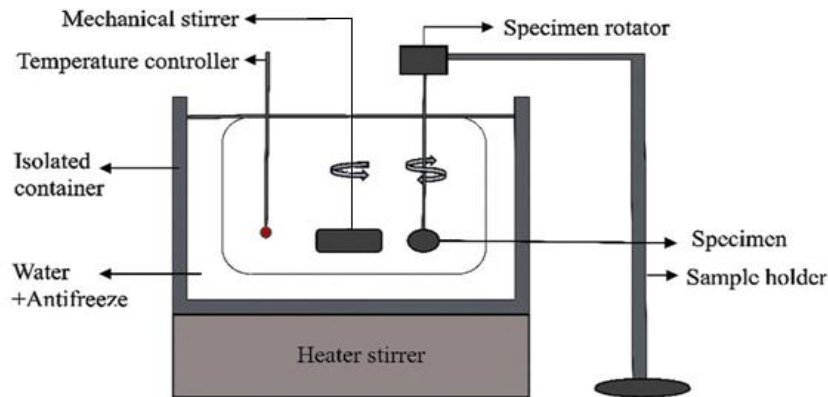
All specimens were polished and grinded. Specimens were immersion in solution include (30g/L Na OH, 60g/L NaCO<sub>3</sub> and 60 g/L NaPbO<sub>4</sub> materials for one minute's period at 70°C temperature with moving the electrolyte with magnetic stirring by supply power (5 volt) caused remove any oil from surface of metal, the samples were dried and then, it directed immersion in solution of coating

### 2.2. Electroless Bath Preparation

After finishing from surfaces preparing for plating, the coating bath of electroless were prepared with concentrations explain in Table (2). Conducted for Ni-B electroless coatings in (1 hour, at 95 ± 1 °C). During coating, solution of the bath was mixed by a magnetic stirrer with decrease of the fluctuations of ionic concentrations. The sample was a monastery in two-way direction every 10 min to produce thickness of uniform coating. Figure (2) experimental part to electroless deposition process.

**Table (2).** Electroless bath conditions.

Bath composition	g/L
(NiCl <sub>2</sub> .6H <sub>2</sub> O -Nickel chloride)	24
( NaBH <sub>4</sub> - Sodium borohydride)	0.8
(EDA -Ethylenediamine (98%))	60 ml/l
lead Nitrate	0.02
(NaOH- Sodium hydroxide)	90
SDS -Sodium Dodecyl Sulfate	2
<b>Condition</b>	
pH	(12-14) OR 13
Temp.	95 °C
Time	1 HR



**Figure (2).** Experimental setup to electroless deposition process.

### 2.3. Heat Treatment and Plasma Nitriding for Plating Samples

Samples (Ni-B) were placed in a (5kW) direct current of plasma to increase PECVD chamber. Heat treatment process was conducted in vacuum furnace for electroless coating samples at 400 °C for 4 hours. Conditions for the plasma-nitrided treatment of electroless bath, was shown at Table (3). In the ending of the process, the specimens were gradually cool in the chamber to arrival the room temperature.

**Table (3).** conditions of plasma nitriding treatment.

Condition	Pluse Dc plasma nitriding (PPN)
Temperature (°C)	400 °C
Pressure	10 <sup>-3</sup> torr
discharge voltage	400-500 V

Frequency	8.9 kHz
Duty cycle	70%
Current(A)	2-3
Gas composition	(50%N <sub>2</sub> -50%H <sub>2</sub> )
Duration (h)	4

## 2.4. Characterization

Microhardness of layers for the coating were calculated with vickers hardness tester, a load (25 g, 15 sec). Then, the average value was taken. Figure (3) show device of vickers hardness tester. Wear test was conducted by Pin-on-disc technique (ASTM G 99 standard). Calculate of specific wear rate was with:  $Ws = w / (lL)$ , which L is the normal load, w is the mass loss and l is the sliding distance. The surface roughness value was calculated coated before and after treatment of heat using parameter Ra in  $\mu\text{m}$ . In this work, Surface roughness was used to Calculated Ra with accuracy 0.05  $\mu\text{m}$ . The coatings were examined for the identification for the crystalline phase. Behavior of corrosion for the coatings was calculated by Tafel extrapolation technique.



Figure (3). Vickers hardness tester.

## 3. Result and Discussion

### 3.1. X-Ray Diffraction

XRD of plating Ni-B explain in Figure (4). XRD for deposited electroless plasma-nitrided Ni-B coating was shown in Figure (5) that an amorphous structure. Amorphous element as boron prevent the nucleation phase of nickel and formation of intermetallic compounds Ni<sub>2</sub>B and Ni<sub>3</sub>B, also boron nitride (BN) [28-30].

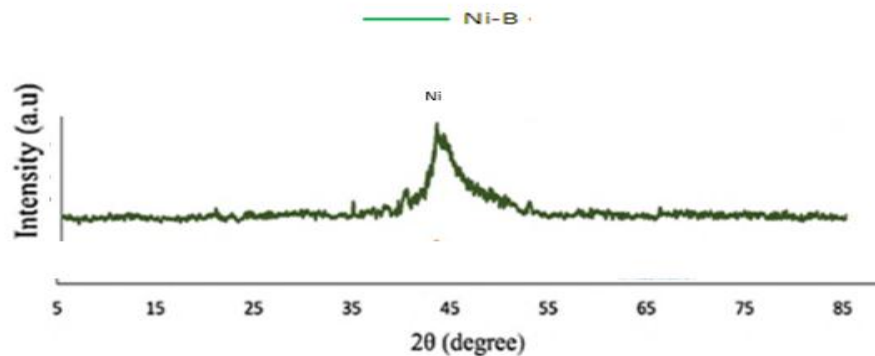


Figure (4). Patterns XRD for plated samples (Ni-B).

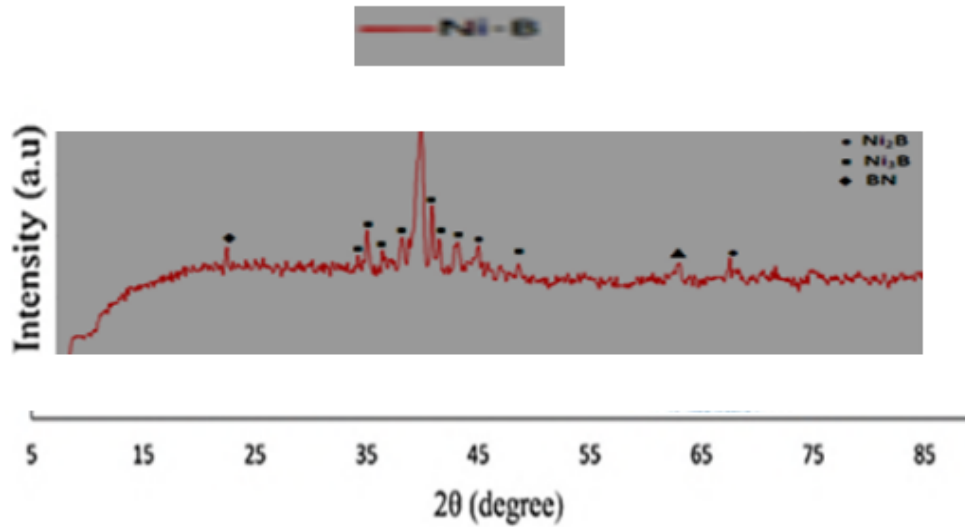


Figure (5). Patterns XRD for plasma nitriding samples Ni-B sample.

### 3.2. Surface Morphology

Figure (6) shows the microstructure of Ni-B coating sample and it is noted that microstructure was a cauliflower type [31, 32].

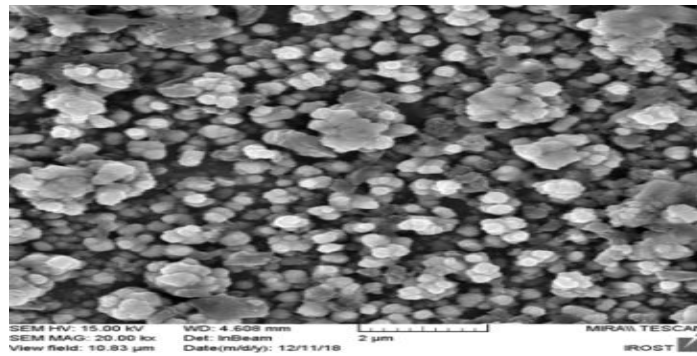


Figure (6). FESEM for electroless coated Ni-B.

Figure (7) FESEM of heat treatment the of Ni-B coating samples show in appearance are matte, also coarseness of microstructure [33].

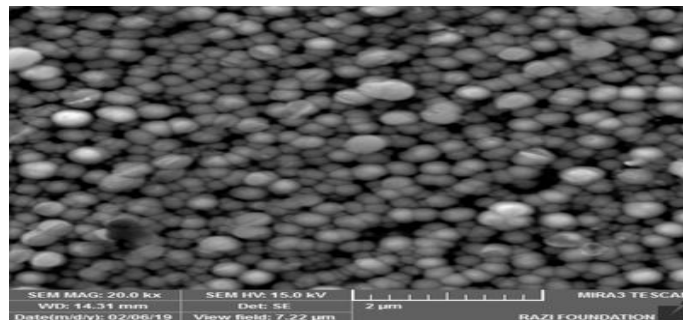


Figure (7). FESEM for heat treated Ni-B.

Figure (8) FESEM of plasma-nitrided of Ni-B coating samples show are change of surface Ni-B to cauliflower microstructure with very clear [31, 34].

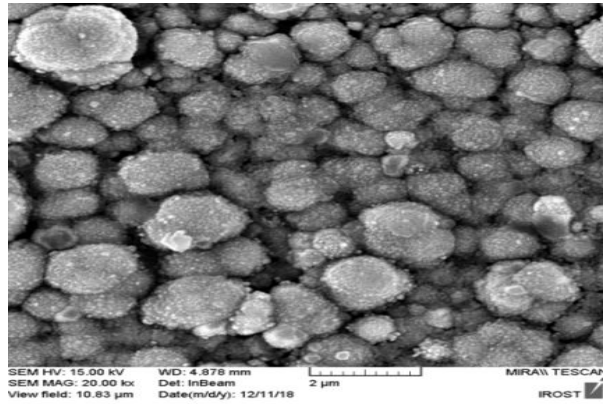


Figure (8). FESEM for plasma-nitrided Ni-B.

### 3.3. Surface Roughness

Data regarding the roughness of the base metal, to plated, annealed and plasma-nitriding specimens has been illustrated in Table (4). At noted, a plasma-nitrided lead to a good increase in roughness and more than the annealing his fact is agreed with [35, 36].

Table (4). Result for samples of surface roughness.

Sample	Ra(μ)
Substrate	0.02
a plated- Ni-B	0.1
annealed- Ni-B	0.26
a plasma-nitrided- Ni-B	0.34

### 3.4 Microhardness

Figure (9) shows microhardness of the samples that underwent the plasma-nitrided and heat treatment. The affects the plasma-nitrided and heat treatment for coating its properties significantly. Hardness are enhanced by heat treatment and the plasma-nitrided. As a noted generally, because of changes of the thermally induced microstructural, and an effect on value of the hardness compared with the value as-deposited state this fact is agree with [37]. The increase of hardness is related with Ni<sub>2</sub>B, Ni<sub>3</sub>B and BN phases [40].

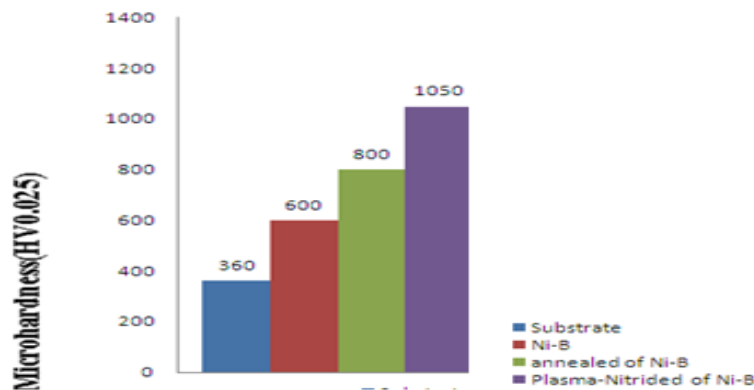
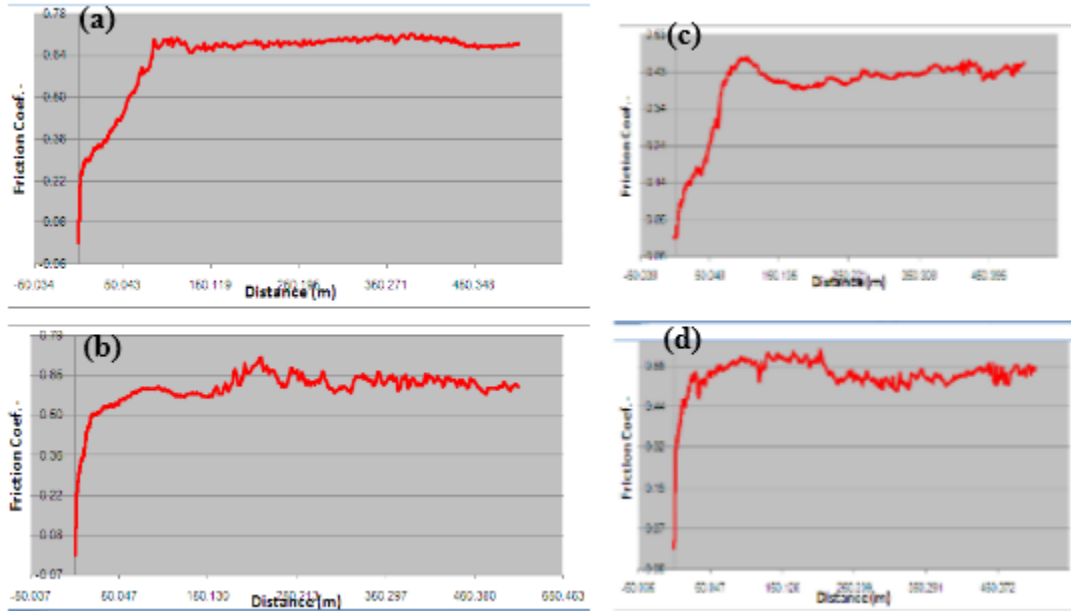


Figure (9). Microhardness of coating, the plasma-nitrided and heat treatment.

### 3.5. Wear Behaviour

Figure (10a) indicates the relationship between distance the friction coefficient for sample of substrate. Figure(10b) indicate the relationship between distance the friction coefficient for samples of as-plated. Those behavior, and amorphous crystallization of a plated specimen during wear test because of generation of the heat which produce at occurrence of stresses of tensile at the interface of amorphous and crystalline phases [26,32,41,42]. Figures (10) indicate the relationship between distance the friction coefficient of plasma nitriding and heat treatment specimen, show was decrease of the friction coefficient. Because, large microhardness value and Ni<sub>2</sub>B, Ni<sub>3</sub>B and BN phases.



**Figure (10).** relationship between distance the friction coefficient (a) substrate. (b) Nickel-Boron, electroless coated samples. (c) Nickel-Boron, annealed samples. (d) Nickel-Boron, plasma nitriding samples.

Table (5) show of rate of specific wear, friction coefficient and mass loss of as-coated, annealed and plasma-nitrided samples. It can be noted in the plasma-nitriding Ni-B has the lesser rate of wear specific which is related by the big mutual solubility of iron and nickel [43, 44].

**Table (5).** Results of rate of specific wear, friction coefficient of samples.

(Sample)	(Coefficient of friction)	(Mass loss (mg))	(Specific wear rate) (g/N.M)x 10 <sup>-4</sup>
Substrate	0.68	1.7	3.4E-4
a plated- Ni-B	0.61	0.5	1E-4
a annealed- Ni-B	0.55	0.45	0.9E-4
a plasma-nitrided- Ni-B	0.45	0.2	0.4E-4

Figure (11) Analysis EDS of worn surface for samples, show to oxygen amount addition in plasma-nitriding samples [45].

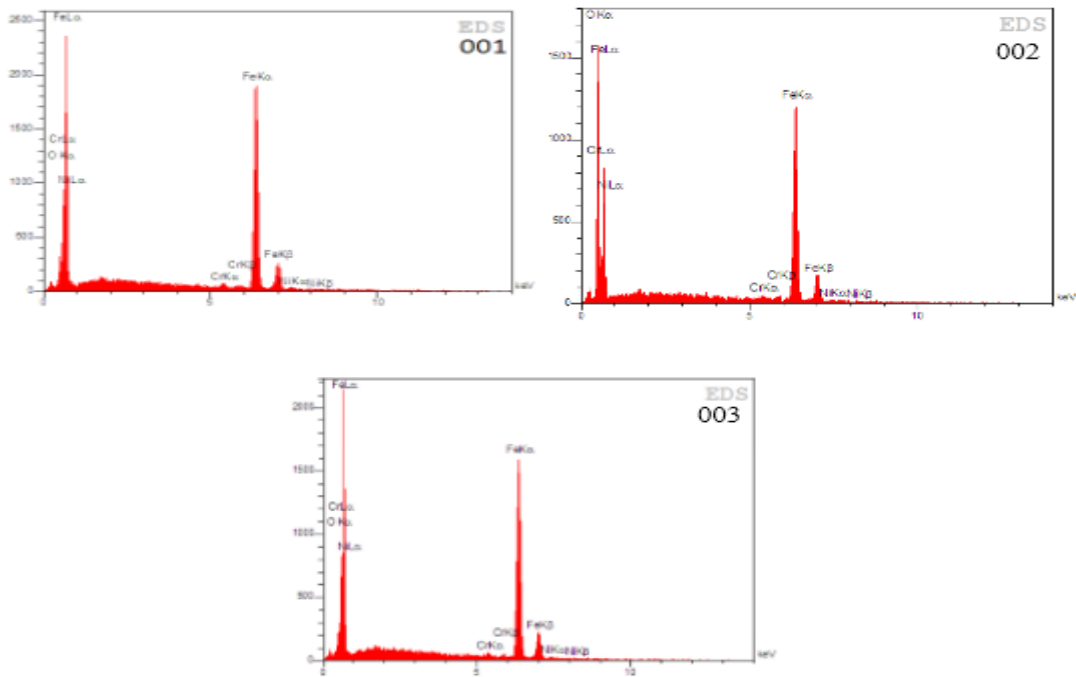


Figure (11). Analysis EDS of worn surface for samples.

### 3.6. Corrosion Behaviour

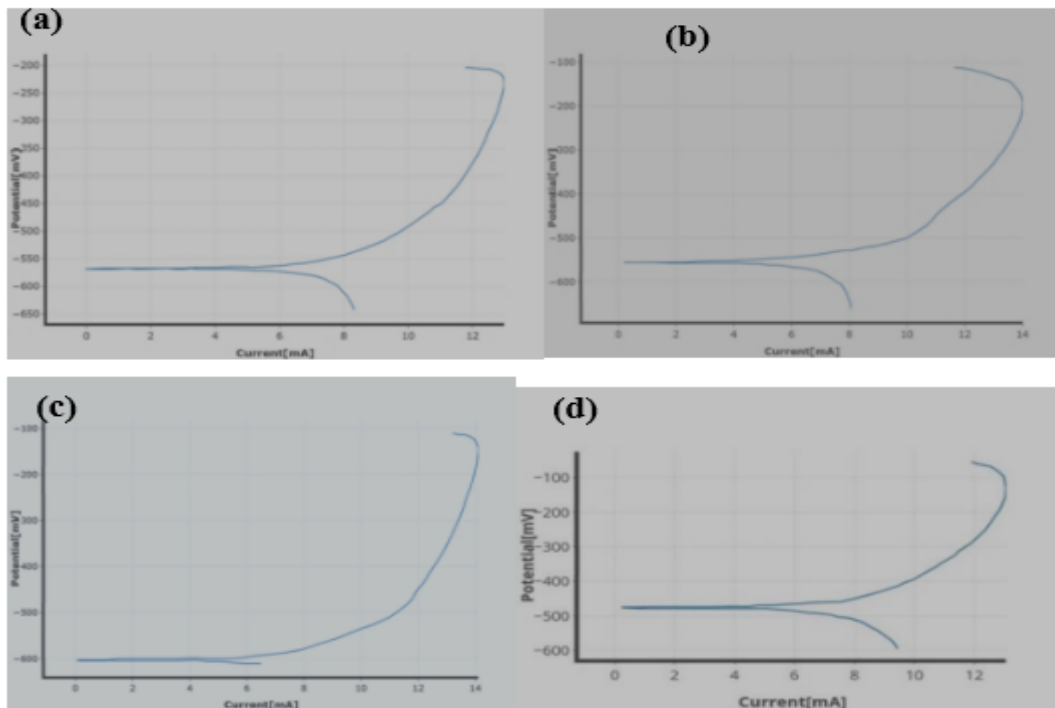
Table (6) include the results of test of electrochemical,  $E_{corr}$ ,  $I_{corr}$ , and corrosion rate coatings, evaluated by Tafel extrapolation technique in solution of 3.5% NaCl.

Table (6). The results of test of electrochemical.

Sample	$E_{corr}$ (mV)	$I_{corr}$ ( $\mu$ A)	Corrosion Rate (mpy)
Substrate	-571	49.49	6.9
(Nickel-Boron)a plated	-551	27.83	4
(Nickel-Boron) a annealed	-606	13	1.4
(Nickel-Boron) a plasma nitriding	-391	9	1

Potentiodynamic polarization curves measured on uncoated and NiB coated 4340 steel are shown on Figure (12). It is clear that the use of a Ni-B coating in enhancement of properties of the corrosion of AISI 4340 steel is due to thickness of electroless deposition is so uniform and the cold of Ni-B coating to act as an active barrier between corrosion media and metal, the value of  $E_{corr}$  is less negative, rate of the corrosion and  $I_{corr}$  is lesser on the plated sample than uncoated 4340steel are shown in Table(6) [46]. Graphic observation of the curves after various treatments are shown in Figure (12), which indicates less negative ( $E_{corr}$ ) values caused by nitrogen diffusion [39].

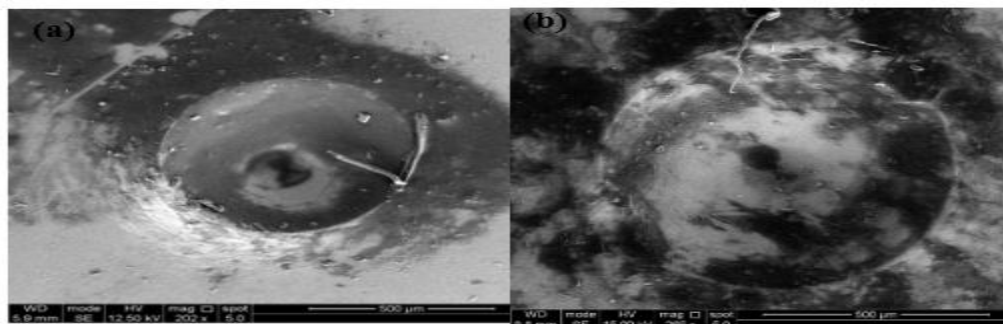




**Figure (12).** polarization of Tafel curves of the (a) as-substrate; (b) Ni-B electroless coated samples. (c) Ni-B, annealed samples. (d) Ni-B, plasma nitriding samples.

### 3.7. Adhesion Test

Figure (13) shows SEM image of Rockwell C on the coating sample. long and deep cracks are noted around the indentation point in the coating sample. These results show that the coating sample has an acceptable adhesion strength, as shown in Figure (12 b), however the substrate has poor adhesion, show in Figure (12 a) [47].



**Figure (13).** Images SEM of Rockwell C effect an indentation on coating sample, (a) substrate and (b) coating.

### 4. Conclusions

the formation of BN and Ni<sub>2</sub>B, Ni<sub>3</sub>B phases by plasma-nitrided of electroless(Ni-B) coating, which increased microhardness. the surface roughness increased, but decreased mass loss with plasma nitride of coatings because the bigger hardness. plasma-nitriding process may be a good replacement for heat treatment. also plasma-nitrided os electroless Ni-B coating, produce diffusion of nitrogen atoms in matrix of Ni and creation of Ni<sub>2</sub>B, Ni<sub>3</sub>B, and BN phases, lead to increase the hardness. The big resistance of wear related with the sample of (Ni-B). These can be associated with the smaller size of grain. Ni-B coating is used in the enhancement of properties of the corrosion of AISI 4340 steel is due to the thickness of electroless deposition, which is uniform, and the cold of Ni-B coating to act as an active barrier between corrosion media and metal, it should be noted that the value of E<sub>corr</sub> is less negative and the rate of corrosion and I<sub>corr</sub> is lower on the plated sample than on the uncoated sample.

## Acknowledgement

The authors hereby acknowledge support from academic stuffs and support stuffs and I would like to thank the staff of chemistry lab. in Al-Mustaqbal University College, Iraq.

## References

- [1] A. R. Boccaccini and I. Zhitomirsky, *Mater. Sci.*, vol.6, pp.251, 2002.
- [2] D. Thiemig and A. Bund, *Surf. Coat. Tech.*, vol. 202, pp. 2976, 2008.
- [3] Z. Liu and W. Gao, *Surf. Coat. Tech.*, vol. 200, pp. 5087, 2006.
- [4] L. p. Wu, J. j. Zhao, Y. p. Xie and Z.d. Yang, *Trans. of Nonferrous Met. Soc. China.*, vol. 2, pp. s630, 2010.
- [5] Riedel A. Electroless nickel plating. *Finishing Publication LTD.*, London, 1989.
- [6] US Patent, vol.2, pp.461-661, 1945.
- [7] P. Sahoo and S. K. Das, *Mater. Design*, vol.32, pp. 1760, 2011.
- [8] Y. Yang, W. Chen, C. Zhou, H. Xu and W. Gao, *App. Nanosci.*, vol.1, pp. 19, 2011.
- [9] S. Afroukhteh, C. Dehghanian and M. Emamy, *Mater. Inter.*, vol. 22, pp.480, 2012.
- [10] C. Li, Y. Wang and Z. Pan, *Mater. Design*, vol. 47, pp. 443, 2013.
- [11] V. Vitry, A. Sens, A. F. Kanta and F. Delaunois, *App. Surf. Sci.*, vol. 263, pp.640, 2012.
- [12] Y. W. Riddle and T. O. Bailerare, *J. Mater Proc. Tech.*, vol. 57, pp.40, 2005.
- [13] T. S. N. Sankara Narayanan, A. Stephan and S. Guruskanthan, *Surf. Coat., Tech.*, vol.179, pp.56, 2004.
- [14] K. Krishnaveni, T. S. N. Sankara Narayanan and S. K.Seshadri, *Surf. Coat., Tech.*, vol. 190, pp. 115, 2005.
- [15] Z. C. Wang, F. Jia, L. Yu, Z. B. Qi, Y. Tang and G. L. Song, *Surf. Coat. Tech.*, vol.206, pp.3676, 2012.
- [16] F. Bülbül, H. Altun, V. Ezirmik and Ö. Küçük, *J. Eng. Tribology.*, pp.1-11, 2012.
- [17] T. p. Xuan, L. Zhang and Q. h. Huang, *Trans. Nonfer. Met. Soc. China.*, vol.16, pp.363, 2006.
- [18] H. B. Hassan, Z. A. Hamid, *Int. J. Hydrogen Energy.*, vol.36, pp.849, 2011.
- [19] V. Vitry, A.-F. Kanta, F. Delaunois, "Application of nitriding to electroless nickel-boron coatings: chemical and structural effects; mechanical characterization; corrosion resistance," *Mater. Des.* vol.39, pp.269-278, 2012.
- [20] Y. He, S. Wang, F. Walsh, Y.-L. Chiu, P. Reed, "Self-lubricating Ni-P-MoS<sub>2</sub> composite coatings," *Surf. Coat. Technol.* vol.307, pp.926-934, 2016.
- [21] Y. Wan, Y. Yu, L. Cao, M. Zhang, J. Gao, C. Qi, "Corrosion and tribological performance of PTFE-coated electroless nickel boron coatings," *Surf. Coat. Technol.* vol.307, pp.316-323, 2016.
- [22] C. Carpenter, P. Shipway, Y. Zhu, "Electrodeposition of nickel-carbon nanotube nanocomposite coatings for enhanced wear resistance," *Wear*, vol.271, pp. 2100-2105, 2011.
- [23] M.-F. Yu, O. Lourie, M.J. Dyer, K. Moloni, T.F. Kelly, R.S. Ruoff, "Strength and breaking mechanism of multiwalled carbon nanotubes under tensile load," *Science*, vol.287, pp.637-640, 2000.
- [24] P.-C. Tsai, Y.-R. Jeng, J.-T. Lee, I. Stachiv, P. Sittner, "Effects of carbon nanotube reinforcement and grain size refinement mechanical properties and wear behaviors of carbon nanotube/copper composites," *Diam. Relat. Mater.* vol.74, pp.197-204, 2017.
- [25] L. Melk, J.J.R. Rovira, M.-L. Antti, M. Anglada, "Coefficient of friction and wear resistance of zirconia-MWCNTs composites," *Ceram. Int.* vol. 41, pp.459-468, 2015.
- [26] Q. Wang, M. Callisti, A. Miranda, B. McKay, I. Deligkiozi, T.K. Milickovic, A. Zoikis- Karathanasis, K. Hrissagis, L. Magagnin, T. Polcar, "Evolution of structural, mechanical and tribological properties of Ni-P/MWCNT coatings as a function of annealing temperature," *Surf. Coat. Technol.* vol.302, pp.195-201,2016.
- [27] Q.Barati ,Seyed Mohammad Mehdi Hadavi " Electroless Ni-B and composite coatings: A critical review on formation mechanism, properties, applications and future trends" *Surfaces and Interfaces* , vol.21, pp. 100702 , 2020.
- [28] Q.-L. Rao, G. Bi, Q.-H. Lu, H.-W. Wang, X.-L. Fan, "Microstructure evolution of electroless Ni-B film during its depositing process," *Appl. Surf. Sci.* vol.240, pp.28-33, 2005.
- [29] E. Georgiza, V. Gouda, P. Vassiliou, "Production and properties of composite electroless Ni-B-SiC coatings," *Surf. Coat. Technol.* vol.325, pp. 46-51, 2017.
- [30] V. Vitry, A.-F. Kanta, F. Delaunois, "Application of nitriding to electroless nickel-boron coatings: chemical and structural effects; mechanical characterization; corrosion resistance," *Mater. Des.*vol. 39, pp.269-278, 2012.
- [31] V. Vitry, L. Bonin, Formation and characterization of multilayers borohydride and hypophosphite reduced electroless nickel deposits," *Electrochim. Acta*, vol.243, pp.7-17, 2017.

- [32] A. Mukhopadhyay, T.K. Barman, P. Sahoo, "Tribological behavior of sodium borohydride reduced electroless nickel alloy coatings at room and elevated temperatures," *Surf. Coat. Technol.*, vol.321, pp. 464-476, 2017.
- [33] S. K. Das, P. Sahoo: *Tribology in industry*, vol.32, pp.17-25, 2010.
- [34] M. Anik, E. Korpe, E. Şen, "Effect of coating bath composition on the properties of electroless nickel-boron films," *Surf. Coat. Technol.*, vol. 202, pp.1718-1727, 2008.
- [35] M. HeydarzadehSohi, M. Ebrahimi, A. HonarbakhshRaouf, F. Mahboubi: *Surf.Coat. Technol.*, vol. 205, pp. S84-S89, 2010.
- [36] G. Prasad Singh, J. Alphonsa, P.K. Barhai, P.A. Rayjada, P.M. Raole, S. ukherjee: *Surf. Coat. Technol.*, vol.200, pp.5807–5811, 2006.
- [37] J. Umeda, B. Fugetsu, E. Nishida, H. Miyaji, K. Kondoh, "Friction behavior of network-structured CNT coating on pure titanium plate," *Appl. Surf. Sci.*, vol.357, pp.721-727, 2015.
- [38] M. Yan, H. G. Ying, T. Y. Ma: *Surf. Coat. Technol.*, vol.202, pp.5909–5913, 2008.
- [39] A. F. Kanta, V. Vitry, F. Delaunois: *Mater. Lett.*, vol.63, pp.2662–2665, 2009.
- [40] V. Vitry, A.-F. Kanta, F. Delaunois, "Mechanical and wear characterization of electroless nickel-boron coatings," *Surf. Coat. Technol.*, vol.206, pp. 1879-1885, 2011.
- [41] L.Y. Wang, J. Tu, W. Chen, Y. Wang, X. Liu, C. Olk, D. Cheng, X. Zhang, "Friction and wear behavior of electroless Ni-based CNT composite coatings," *Wear*, vol. 254, pp. 1289-1293, 2003.
- [42] V. Puchy, P. Hvizdos, J. Dusza, F. Kovac, F. Inam, M. Reece, "Wear resistance of Al<sub>2</sub>O<sub>3</sub>-CNT ceramic nanocomposites at room and high temperatures," *Ceram. Int.*, vol. 39, pp.5821-5826, 2013.
- [43] K. Krishnaveni, T. S. N. Sankara Narayanan, S. K. Seshadri: *Surf. Coat. Technol.*, vol. 190, pp.115–121, 2005.
- [44] M. Palaniappa, S. K. Seshadri: *Wear*, vol.265, pp. 735–740, 2008.
- [45] M.M. Bastwros, A.M. Esawi, A. Wifi, "Friction and wear behavior of Al-CNT composites," *Wear*, vol.307, pp. 164-173, 2013.
- [46] Kanta AF, Poelman M, Vitry V, Delaunois F. "Nickel–boron electrochemical properties investigations". *J Alloy Compd*, vol. 505, pp.151–6, 2010.
- [47] Yusuf Kayali & Sukru Taktak, "Characterization and Rockwell-C adhesion properties of chromium-based borided steels". *Journal of Adhesion Science and Technology*, Vol. 29, pp. 2065-2075, 2015.