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Influence of Incorporating B₄C Nanoparticles and Pulse Electrodeposition Parameters on the Surface Morphology and Wear Behavior of Nickel Based Nanocomposite Coatings

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ABSTRACT

The main aim of this project is to fabricate a nickel-based nanocomposite coating reinforced with B₄C nanocomposite coating by the means of pulse electrodeposition technique in nickel sulfate bath under high deposition rate and study the impact of pulse parameters on surface morphology and tribological properties. The coating surface morphology, chemical composition, microstructure, microhardness, surface topography, and ultimately wear behavior of coatings were assessed through scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffraction, Vickers hardness, atomic force microscopy, and pin-on-disk wear test, respectively. By the incorporation of B₄C nanoparticles into the nickel matrix, the surface morphology was altered from pyramid to nodular structure. The maximum B₄C incorporation was recorded to be 6.45 vol.% at 10 A/dm², γ = 50%, and 10 Hz. The crystallite size of nickel crystallographic planes (111) and (200) reduced, so was the grain size. The microhardness of all composite samples was significantly enhanced. The weight loss and wear rate values of Ni-B₄C composites were diminished and the minimum weight loss in the form of weight gain was dedicated to Ni-B₄C sample fabricated at 10 A/dm², by the -0.23 mg amount. The friction coefficient and average roughness were significantly increased. Notably, the wear morphology indicated the dominancy of adhesive wear specially in pure nickel sample, but it turned out to be abrasive wear for almost all Ni-B₄C samples in which the its reduction was associated with the decrement of adhesion, plastic deformation, delamination, ploughing, and also a bit abrasive type of wear. Overall, the Ni-B₄C sample fabricated at 10 A/dm², γ = 50%, and 10 Hz, experienced the optimum wear resistance.

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1. INTRODUCTION

From a long time ago, dip-coating, sol-gel, spincoating, layer-by-layer method, mechanochemical technique, thermal spraying, sol-gel, biomimetic, dynamic chemical plating, electrophoretic deposition, and electrodeposition, also called "electroplating", have been regarded as a fundamental electrochemical technique for fabricating thin films, alloy, and composite coatings in order to improve corrosion and mechanical properties such as hardness, surface morphology, and tribology [1-28]. Among these techniques, electrodeposition has been extensively employed for fabricating pure base metal and composite micro and nano coatings. Electrodeposition is regarded as a rather easy and low-cost method for deposition of coatings, in which the initial size of the nuclei can be controlled by the applied overpotential leading to dense and non-porous coatings [29,30]. Another important advantage of this technique is related to its capability of implementing vast range of metallic elements in coatings. In electroplating, owing to the presence of electric current in the

electrolyte solution, the metal ion adsorbs electrons in the electrolyte and deposits on the cathode (positively charged) surface. As a result of this electric current between anode (negatively charged) and cathode, an electric field is created and facilitates the movement of anions (negatively charged ions) and cations (positively charged ions) toward the anode the cathode, respectively, resulting on coating formation on the cathode surface [31,32]. The power supply in this method, can convert the alternative current (AC) to direct current (DC) and provide the required electric charge for electrodeposition [33]. Possessing outstanding properties of nickel element including corrosion resistance, wear resistance, high hardness, electrical conductivity, and remarkable flexibility makes it highly applicable in various industries [31, 33-34]. Notably, the significant adherence, contact resistance, and high corrosion resistance of nickel coating in aqueous, alkaline, organic, and mineral acids due to formation of a passive layer, indicates the usefulness and importance of applying nickel

as the base metal in metal matrix coatings (MMCs) [35]. The electroplating of nickel is recognized as the most important techniques in industries as more than half of the parts fabricated of nickel coating [4, 31, 33-39]. In the process of nickel plating, two electrodes (anode and cathode) are rinsed in the conducting solution with nickel salts, and a DC current is applied leading to solving anode surface and forming nickel coating on cathode as a result of electrons adsorption to positive ions at the cathode surface [31,38,40]. Pulsed electrodeposition (PED) technique as a powerful alternative DC electrodeposition method creates a great chance to control the electrodeposits parameters and the microstructure as well as the mechanical and corrosion properties of the electrodeposited In electrodeposition, coatings. РС pulse parameters such as current density, duty cycle, and pulse frequency can highly influence the electrodeposits morphology leading to alteration in the hardness, surface morphology (smooth or nodular), texture, microstructure, incorporation, and agglomeration of particles that ultimately influence the surface topography and tribological properties [35,41-44]. There are numerous researches in literature indicating the improved microstructure, texture, corrosion, hardness, and in particular, tribological properties of nickelbased coatings, as a result of embedding secondary particles into the based coating [4,31,33-34,44-49]. Zhang et al. [50] fabricated a Ni-B/B₄C composite coating on N80 steel via pulsed electrodeposition (PED) in which average hardness and friction coefficient in Ni-B/B₄C over the Ni-B coating. In another research, a B₄C-based coating was produced by Zhu et al. [51] via Vacuum Plasma Spray (VPS) technique using electroless plating composite powders comprised of B₄C and 10 and 20 vol.% of Ni, the results indicated the noticeable enhancement of wear resistance in the case of participating 10 vol.% Ni into coating matrix demonstrated the optimum tribological properties and the dominant wear

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mechanism was oxidation and a transfer layer created on the worn surface. Seldom systematic research has been dedicated to the assessment of reinforced Ni-B₄C coating. Paydar *et al.* [52], proved the increased wear resistance of Ni-BN-B₄C compared to pure Ni coating. In another research conducted by Shrestha et al., the participation of azobenzene surfactant (AZTAB) not only played as a dispersing agent in the bath, but also acted as a promoter to embed high amount of B₄C particles (45 vol.%) into nickel matrix due to its more positive reduction potential than that of the nickel [53]. The novelty of this work is to investigate the electrodeposition characteristics and tribological behaviour of nickel-B₄C nanocomposite coatings produced by pulsed current electrodeposition through studying the effect of changing the pulse parameters such as current density, duty cycle, and pulse frequency of the coating.

2. EXPERIMENTAL

In this project, with the aid of pulse electrodeposition, Ni-B₄C nanocomposite coating was successfully deposited on the copper base plate. Afterwards, the main properties of the coating were assessed via surface morphology using scanning electron microscopy (SEM), chemical composition of the coating via energydispersive x-ray spectroscopy (EDS), microstructure through x-ray diffraction (XRD), surface morphology by atomic force microscopy (AFM), microhardness, and finally the wear behaviour of the coating samples were determined by the Pin-on-Disc method. Just before electrodeposition, the copper base plates were mechanically polished to obtain mirror-like surface finish, and then the samples were exposed to acetone and acid washed in 10% HCl solution to activate sample surface to be free of grease and any other pollution, and finally were rinsed in distilled water followed by drying with hot air before any further tests. After the preparation of copper surfaces, the samples had to be

electrodeposited instantly. The cathode with the surface area of 20 mm×20 mm was to be rinsed in the solution and attached to the negative pole, and the anode to the positive pole. The distance between anode and cathode were 2 cm and a specified time was set for electrodeposition. The main electrolyte solution composition was constituted of 40 g/l boric acid (H₃BO₃), 300 g/l nickel sulfate NiSO₄.6H₂O, and 40 g/l nickel chloride $(NiCl_2.6H_2O).$ Afterwards, B₄C nanoparticles with the size of less than 50 nm were added to the solution to embed into the nickel matrix coating, and then sodium dodecyl sulfate (SDS) was also added to the electrolyte. SDS is an anionic surfactant that once adsorbed on the particles surface, improves the suspension stability by enhancing the electrostatic repulsion between particles and by altering the wettability of the suspended particles. The solution was then stirred up to 24 hours in the environment temperature and was fully re-disperse in an ultrasonication process for one hour with the stir rate of 250 rpm and 50 °C to prevent their agglomeration during electrodeposition and obtain a homogenous distribution of nanoparticles. The Ni-B₄C nanocomposite coating sample was electrodeposited at $i_p = 7 \text{ A/dm}^2$, $\gamma =$ 50%, f = 10 Hz, and was chosen to be the baseline coating sample for the whole experiments.

3. Results and Discussion

3.1. Chemical Composition of Ni-B4C Nanocomposite Coatings via EDS

As mentioned earlier, the presence of electrodeposited nanoparticles in Ni-B₄C coating is determined by EDS elemental analysis. In Fig 1, the elemental analysis and distribution map of Ni-B₄C sample as the baseline sample deposited at the current density of 7 A/dm², duty cycle of 50%, and pulse frequency of 10 Hz, is clearly observed.



Fig 1.Carbon map distribution of the Ni-B4C baseline nanocomposite sample at 7 A/dm², duty cycle of 50%, and f=10 Hz





3.2. Incorporation Rate of B4C Nanoparticles under Different Pulse Variants

According to Fig 2, by increasing the current density from 7 to 10 A/dm², the incorporation rate of B₄C nanoparticles significantly rises from 5.25 to 6.45 vol. %, describing the fact that by the rise of current density, the driving force of Ni²⁺ ions reduction and their mobility have increase in the solution resulting in the enhancement of B₄C nanoparticles participation.

As observed in Fig 2, by the rise in duty cycle from 50 to 75%, the incorporation rate of B_4C nanoparticles has enhanced from 5.25 to 6 vol.%, as described in Equation (1):

$$i_A = i_p \times \gamma$$
 (1)

 $i_{A:}$ Average current density, $i_p:$ Peak current density, γ = duty cycle

The variation in duty cycle is proportional to variation of i_A which leads to the enhancement of reduction rate of Ni²⁺ ions in the electrolyte, and

thus the rise in incorporation rate of B_4C nanoparticles in the coating matrix.

By raising the pulse frequency from 10 to 100 Hz, the participation rate of B_4C nanoparticles has increased from 5.25 to 5.9 vol. % (see Fig 2). As a matter of fact, according to Equation (2):

$$f = \frac{1}{T} = \frac{1}{T_{ON} + T_{OFF}} \tag{2}$$

f: Pulse frequency, T_{ON} : Current On, T_{OFF} : Current Off

By increasing the frequency, T ($T_{ON} + T_{OFF}$) is decreased and, in constant deposition time, the total electrodeposition cycles have risen which results into the more opportunity for B₄C nanoparticles to be incorporated in the matrix of Ni coating.

It is concluded that the composite coatings with the highest amount of evenly distributed particle incorporation exhibit superior tribological properties (e.g., wear resistance) which is the expected classic behavior and is widely reported in literature [32,41,45,54].

3.3. Morphological Studies after Electrodeposition and before Running Wear Test

The morphological studies of the pure nickel and Ni-B₄C nanocomposite base sample, illustrated in Fig 3, indicates that by incorporating B₄C nanoparticles into the matrix, the morphology changes from a fine pyramid form to an agglomerated structure. As a matter of fact, by embedding B₄C nanoparticles in the coating, the morphology is varied from pyramid form (see Fig 3(a)) to agglomerated - nodular structure (see Fig 3(b-e)) and it is also evident from the literature that the nodular (or spherical) structure is rougher than the pyramid structure belonging the pure nickel sample [33,36,55]. All the Ni-B₄C (Fig 3(b-e)) samples illustrate a nodular (spherical) structure and rougher surface. Moreover, the size of these nodules differs with the alteration in electrodeposition parameters such as current density, duty cycle, and pulse frequency and these variations in morphology are originated from the fact that the variation in incorporation rate of B₄C nanoparticles is highly under impression of various pulse electrodeposition variants. At the maximum incorporation rate of B₄C nanoparticles (6.45 vol.% - see Fig 2), the most nodular and agglomerated microstructure is observed in the SEM micrograph of Ni-B₄C sample at $i_p = 10$ A/dm², γ = 50%, and f = 10 Hz.



Fig 3.The SEM micrographs of pure nickel and Ni-B₄C composite coatings after electrodeposition at the baseline sample state condition: $i_p = 7 \text{ A/dm}^2$, $\gamma = 50\%$, f = 10 Hz. (a) Pure nickel, (b) Ni-B₄C, (c) Ni-B₄C, $i_p = 10 \text{ A/dm}^2$, (d) Ni-B₄C, $\gamma = 75\%$, and (e) Ni-B₄C, f = 100 Hz

3.4. Surface Topography of Ni-B4C Nanocomposite Coatings via AFM

The two-dimensional surface topography of the samples and also the calculated mean roughness with respect to each sample via the atomic force microscopy (AFM) is depicted in Figures 4 and 5. According to Fig 4, by B_4C participation in the Ni matrix, the surface topography becomes rougher than that of pure nickel sample. As observed in Fig 5, the pure nickel sample possessed the minimum mean surface roughness R_s of 0.16 µm, but by incorporation of B_4C nanoparticles into the nickel matrix, the mean surface roughness has increased

and reached its peak for Ni-B₄C sample deposited at 10 A/dm² with the maximum incorporation rate of 6.45 vol.% (see Fig 2).

Besides, by rising the surface roughness, surface that are already touched to each other would be more in touch and more wear rate is expected; however, the incorporation of B₄C nanoparticles has led to increase in microhardness so that prevented the surface layers to be peeled off, thus despite the higher roughness in the Ni-B₄C samples, the wear resistance has magnificently improved in Ni-B₄C samples with respect to that of pure nickel sample.







Fig 5.The calculated mean roughness obtained from AFM data analysis (demonstrated in Figure 4) under different pulse parameters

3.5. Microstructure of Ni-B4C Nanocomposite Coatings via XRD

The results of data analysis attained from X-Ray Diffraction (XRD) analysis under various pulse variants. The crystallite size of nickel coating is also derived using Scherrer's relationship (Equation (3)):

$$D = \frac{K\lambda}{\beta \cos \theta}$$
(3)
Where,

D: Crystallite size, K: Shape factor Constant, λ : The X-ray wavelength in nm, β : the width of the peak at the half intensity or FWHM ($\frac{FWHM}{Radians}$), θ : The diffraction angle in radians.

Fig 6 describes the XRD pattern under various pulse electrodeposition parameters for pure nickel and Ni-B₄C nanocomposite samples. As depicted, a tiny little peak indicates the crystal plane of B₄C nanoparticles (021) on each XRD pattern of all Ni-B₄C samples. It is evident from Table 1 and 2, and Fig 6 that the main crystal planes of (111) and (200) are the main planes that by varying pulse parameters compete with each other. By incorporation of B₄C nanoparticles into the matrix, the crystallite size of these planes in all Ni-B₄C samples has reduced which would be due to the rise of nucleation sites and reduction in the crystallite size of nickel base matrix as a result of B₄C nanoparticles incorporation and also alterations in the pulse variants.



Fig 6.The results of XRD data analysis under various pulse parameters

Ni and Ni-B ₄ C nanocomposite coatings under different pulse variants			
Sample	Crystallite size (nm)	Crystallite size (nm)	
	(111)	(200)	
Pure Nickel (Baseline)	36.34	37.41	
Ni-B ₄ C (Baseline)	34.12	28.13	
Ni-B ₄ C (i: 10 A/dm ²)	34.13	23.41	
Ni-B4C (f: 100 Hz)	30.26	25.54	
Ni-B4C (γ: 75%)	34.17	23.44	

Table1.The calculated crystallite size obtained from XRD analysis for pure Ni and Ni-B₄C nanocomposite coatings under different pulse variants

3.6. Tribology and Wear of Ni-B4C Nanocomposites

For a better understanding of tribology and wear of Ni-B₄C samples, various influential parameters were assessed to ultimately study and discuss the wear behaviour of pure nickel and nanocomposite samples. These parameters are as follows:

i) Microhardness of coating samples, ii) Weight loss after Pin-on-disc test, iii) Wear rate of coating samples after Pin-on-disc test, iv) Friction coefficient corresponding to variant pulse parameters, and v) Morphology after wear.

3.6.1. Microhardness

According to Fig 8, by incorporation of B_4C nanoparticles into the Ni matrix, all Ni-B₄C samples under various pulse parameters experience significant rise in microhardness compared to that of pure Ni, indicating the remarkable and effective participation of B₄C nanoparticles into the matrix. As clearly stated earlier in the XRD analysis results in section 3-5, B₄C incorporation results in more nucleation and reduction in crystallite size so that the microhardness has risen as a result. Notably, by increasing the three effective pulse variants (current density, duty cycle, and pulse frequency), the incorporation of B₄C nanoparticles was increased so was the microhardness and showed a similar trend (see Fig 2 and 7). However, based on Fig 7, the microhardness of Ni-B₄C baseline sample was 340 HV, by rising the current density from 7 to 10 A/dm^2 the microhardness was noticeably decreased to 290 HV. After enhancing the duty cycle from 50 to 75 %, the microhardness was significantly lowered to its minimum among Ni-B₄C samples touching 260 HV, and finally by rising the pulse frequency from 10 to 100 Hz, it was scarcely reduced to 330 HV compared to that of baseline sample. The similar trend was also observed for Ni-ZnO nanocomposite coatings [41]. However, the maximum microhardness was observed under current density of 10 A/dm². Despite the rise in incorporation rate of B₄C nanoparticles by rising the three pulse parameters, all of these noticeable reduction in microhardness occurred which is highly relied on the changes occurring in the growth of nickel coating different crystal planes due to participation of B₄C nanoparticles. As mentioned in section 3-5, the crystal orientation of atoms in the two main planes (111) and (200), and the peak intensity ration I (111) / I (200) varies with changes in pulse parameters (see Table 2). Nickel crystal structure is FCC (face centred cubic), in which (111) plane is the most compact plane, and the compactness of (200) plane is also lower than (111) plan, so the more atoms arrange in this plane direction, the more microhardness is achieved. All in all, according to Table 2 and Fig 7, in spite of the rise in maximum current density, duty cycle and pulse frequency, and the enhancement of B₄C incorporation, I (111) / I (200) has reduced leading to diminishment of microhardness as a result of arranging more atoms in less compact plane of (200) instead of more compact plane of (111).

Table 2. The peak intensity ratio of (111) and (200) main crystal planes for all Ni-B ₄ C nanocomposites under
different pulse variants

Sample	I (111)/I (200)	
Ni-B4C (Baseline)	3.09	
Ni-B ₄ C (i: 10 A/dm ²)	2.84	
Ni-B ₄ C (f: 100 Hz)	3.22	
Ni-B₄C (γ: 75%)	3	



Fig 7.Microhardness of pure Ni and Ni-B4C samples under different pulse variants

3.6.2. Weight Loss

According to Fig 8, the weight loss of all pure Ni and Ni-B₄C nanocomposite samples after conducting pin-on-disk method is reported under different pulse electrodeposition conditions. As seen, the weight loss of Ni-B₄C samples is remarkably lower than pure Ni sample, which is highly affected by the higher incorporation and microhardness of nanocomposite samples. The minimum weight loss belonged to the Ni-B₄C sample with -0.23 mg; deposited at 10 A/dm^2 which, in fact, weight gain was observed instead of weight loss. To be more precise, the most incorporation rate of B₄C nanoparticles (6.45 vol.%, see Fig 2) in protecting the coating against wear, and also the minimum weight loss (the most wear resistance) indicate the peeled off surface layers due to local plastic deformation occurred

during pin-on-disk test which have been remained on the surface and oxidized and thus a weight gain is observed. By increasing the duty cycle from 50 to 75%, the weight loss has reduced from 0.65 to 0.4 mg meaning an improvement in wear resistance as a result of enhancement in incorporate rate of B₄C nanoparticles from 5.25 to 6 vol. %. Surprisingly, it was observed that by rising the pulse frequency from 10 to 100 Hz, the weight loss rose from 0.65 to 1 mg that could be originated from the fact that increasing the incorporation rate of B₄C nanoparticles from 5.25 to 6 vol.%. Corresponding to frequency rise from 10 to 100 Hz cannot necessarily guarantee the effective participation of B₄C nanoparticles into the nickel matrix. Another reason could be the scarce reduced microhardness and incorporation rate compared to the Ni-B₄C samples fabricated at 7 A/dm², 10 A/dm², and γ =75%.



Fig 8. The weight loss of pure Ni and Ni-B₄C samples under various pulse electrodeposition conditions

In high pulse frequencies, there is less chance for the reinforcing B₄C nanoparticles to reach the double layer and to incorporate in the coating matrix. As a result, the microhardness and wear resistance of the coating are reduced. Totally, according to the literature, it is widely accepted that a higher weight loss is always related to a lower microhardness since during wear more material is removed from softer surfaces, for instance in Ni-Al₂O₃ and Ni-Si₃N₄ nanocomposite coatings [4,34].

3.6.3. Wear Rate

Since the assessment of the wear rate is the most important measured and calculated parameter in investigating the wear behaviour of materials, the Holm-Archard equation plays the crucial role, as follows:

$$k = \frac{Q \times H}{w \times L} \tag{4}$$

Where, H: Hardness (kgf/mm²), W: Applied vertical force (kgf), L: The sliding distance (m), K: Dimensionless wear coefficient,

Q: Wear volume (mm³) [56]. Using the following relationship, we can derive the term Q (wear volume):

$$Q = \frac{\text{Weight loss (g)}}{\frac{((\text{Incorporation rate (\%)} \times B_4C \text{ density}) + ((100 - \text{Incorporation rate (\%)}) \times \text{Nickel density}))}{100}}{(5)}$$



Fig 9.The wear rate of pure Ni and Ni-B₄C samples under various pulse electrodeposition conditions

Finally, the wear rate is derived via the following equation:

Wear rate (W) (× $10^{-4} \text{ mm}^3/\text{N.m}$) = $\frac{\text{Q}}{\text{w} \times \text{L}} = \frac{\text{K}}{\text{H}}$ (6)

As a matter of fact, the wear rate equals to the wear volume under a specified applied load and sliding distance. Fig 9 reports the wear rate amounts of all pure Ni and Ni-B₄C nanocomposite samples. By assessing the trend of wear rate variations of all samples, it is totally in line with that of weight loss variations (see Fig 8 and 9) indicating the impact of B₄C nanoparticles participation in the nickel matrix. What is more, the least wear rate was found to be -0.552×10^{-4} mm³/Nm. Thus, the best wear resistance belonged to the Ni-B₄C sample deposited at 10 A/dm² possessing the incorporation rate of 6.45. vol % (see Fig 2 and 9).

3.6.4. Friction Coefficient

The friction coefficient map in Fig 10 describes the friction coefficient recorded over 100 m sliding distance for pure nickel and all Ni-B₄C nanocomposite coatings. As illustrated, at the first sliding distances from 0 to about 10 m, the friction

coefficient grows from low to high degrees due to the formation of oxide layers and surface pollution. However, by the gradual increase in the sliding distance, these surface pollutions are diminished and a rather smoother surface is appeared. This phenomenon comes from the involvement of worn particles and enhanced friction coefficient between surfaces (coating surface and pin) and in the following since these particles amount does not change anymore, the maximum friction coefficient remains nearly constant and only fluctuates between a confined range or a stable condition where the mean friction coefficient is measured and reported in Fig 11. According to Fig 11, the minimum mean friction coefficient 0.915 belongs to the pure nickel coating. However, by incorporation of B₄C nanoparticles into the nickel matrix, the friction coefficient has risen and reaches to its maximum for the baseline sample fabricated at 7 A/dm², γ = 50%, and f = 10 Hz. Despite the participation of B₄C nanoparticles in the nickel matrix and its influence on the reduction in weight loss, wear rate and increment of microhardness, the friction coefficient has increased due to the effect of these B₄C nanoparticles on morphology of all Ni-B₄C coatings (see Fig 3), since by B₄C incorporation the morphology has turned from a pyramid form to a nodular (spherical) structure. In other words, the nature of morphology in spherical structure of Ni-B₄C with large size (nodular) is rougher and thus possesses higher friction coefficient compared to that of pyramid structure in pure nickel coating [55]. According to Fig 11, the minimum friction coefficient among all Ni-B₄C coatings was recorded to be 1.255 for the Ni-B₄C sample deposited at 10 A/dm² with incorporation rate of 6.45 vol.% B₄C, and the maximum friction coefficient was 1.594 belonging to the Ni-B₄C baseline sample produced at 7 A/dm² with the

incorporation rate of 5.25 vol% B_4C among all Ni-B₄C coatings indicating the fact that by increase in B₄C incorporation into the nickel matrix, the friction coefficient has been lowered confirming the previous results on improvement of wear resistance as a result of B₄C participation in the nickel matrix. Interestingly, the friction coefficient map trend for all nanocomposite coatings was totally in line with the hardness variations (see Fig. 7 and 10), which can be initiated from the fact that incorporation of B₄C nanoparticles in the coatings leads to roughening the surface (see Fig 5), thus the nanoparticles get involved more and more during the wear.







Fig 11. The mean friction coefficient values under different pulse electrodeposition variants

3.6.5. Morphology after Wear

One of the most applied methods to distinguish wear mechanism is the study of morphology after wear. The most important wear mechanisms in metal-on-metal sliding systems are as follows:

i) Adhesive wear, ii) Abrasive wear, iii) Corrosion wear, and iv) Surface fracture Wear [56].

In which the first two ones, adhesive and abrasive wears are of greater importance with respect to the other types. The other principal forms of wear, namely corrosive and surface fracture wear, do not follow equations (4 and 6), but in many cases, they can be assigned equivalent wear coefficients based on prevalent observed wear rates [56]. According to Equations 4 and 6, dimensionless wear coefficient (*K*) is regarded as a pivotal factor in determining the wear and sliding mechanism of surfaces. In the metal-on-metal sliding systems, based on the *K* value, various mechanisms could occur. In this research, *K* values were found to be between the range of 10^{-3} to 10^{-4} , indicating the

probable mechanism of adhesive/abrasive wear. Since the maximum reported *K* values in this study belonged to the pure nickel sample in particular and also the Ni-B₄C sample deposited at higher pulse frequency f = 100 Hz, the possible mechanism for these coatings is closer to adhesion rather than abrasion mechanism. Decrement of K value is indicative of improved wear behavior. Incorporation of B₄C into the matrix and increase in current density reduce the K value and increase the contribution of abrasive mechanism in the wear of the composite coatings. In an abrasive mechanism, the hard reinforced B₄C nanoparticles cause the separation of surface layers and the abrasion of nanoparticles to pin (or any other abrasive material) leads to the separation of nanoparticles by cutting through them or creating grooves [8,57].

According to the morphological micrographs in Fig 11 (a-e), specific signs of adhesive wear appeared as a whole and also a bit abrasive wear

under different pulse variants that are clearly pointed on each sample micrograph. According to the Fig 11a, cracks, plastic deformation, adhesion, delamination, and also noticeable cracks are observed that indicate the occurrence of adhesive wear. It could be mentioned that by incorporating B₄C nanoparticles in the Ni matrix, adhesive wear is reduced, and thus improved the wear resistance as it is totally confirmed in Fig 12(c) for the Ni-B₄C sample fabricated at 10 A/dm², γ = 50%, and f = 10 Hz possessing the best wear resistance among all coatings - in which local plastic deformation and seizure of wear debris is observed indicating the peeled off surface layers from the coating due to the local plastic deformation occurred during pin-on-disk method, and thus these worn particles and oxides on the surface were the crucial sign of weight gain on this sample and not weight loss. As a matter of fact, the dispersion hardening associated with the increased incorporated B₄C nanoparticles in the coating deposited under higher electrodeposition current density led to minimize the adhesion, abrasive grooves, and delamination compared to pure nickel. Meanwhile, plastic deformation and seizure of wear debris due to plastic deformation hardening takes place as observed in our previous work for Ni-ZnO nanocomposite coatings [36]. Furthermore, by enhancing the duty cycle from 50 to 75%, minor traces of delamination, plastic deformation, and ploughing is observed, and by rising the pulse frequency from 10 to 100 Hz, in particular for the Ni-B₄C sample deposited at f =100Hz in which the growth of adhesion, plastic deformation, ploughing, and also seizure of wear debris was accompanied to increased wear rate.



Fig 12. The morphology after wear via SEM for pure nickel and Ni-B₄C composite coatings after electrodeposition at the baseline sample state condition: $i_p = 7 \text{ A/dm}^2$, $\gamma = 50\%$, and f = 10 Hz. (a) Pure nickel, (b) Ni-B₄C, (c) Ni-B₄C, (i) Ni-B₄C, (c) Ni-B₄C,

4. Conclusion

(1) The incorporation of B₄C nanoparticles into the nickel matrix was successfully conducted and confirmed via EDS analysis. The Ni-B₄C nanocomposite coating sample was electrodeposited at $i_p = 7 \text{ A/dm}^2$, $\gamma = 50\%$, and f = 10 Hz and was chosen to be the baseline coating sample for the tests of the projects. According to the SEM micrographs, the morphology of nickel coating after electrodeposition and before wear test was pyramid form and by incorporation of B₄C nanoparticles into the nickel coatings matrix, the morphology has turned into a nodular (spherical) structure. The XRD analysis results indicated the B₄C participation in the nickel matrix owing to appearance of (021) peak on XRD map, and the reduction of crystallite size for (111) planes. and (200) crystal AFM results demonstrated the higher mean rougher surface for Ni-B₄C samples in which the lowest mean roughness for the pure Ni sample appeared to be 0.16 μ m and for the Ni-B₄C sample was found to be the highest amount of 2.251 $\mu m.$

(2) The incorporation rate of B₄C nanoparticles in the nickel matrix was found to be 5.25 vol.% in the baseline sample electrodeposited at 7 A/dm², $\gamma = 50\%$, and f = 10 Hz, and also the microhardness of all Ni-B₄C nanocomposite samples significantly increased with respect to the pure Ni sample.

(3) The weight loss and wear rate of all Ni-B₄C samples were remarkably lower than that of pure Ni sample. Notably, the minimum weight loss and wear rate belonged to the Ni-B₄C sample deposited at $i_p = 10 \text{ A/dm}^2$, $\gamma = 50\%$, and f = 10 Hz, with the amounts of -2.3 mg and -0.552×10⁻⁴ mm³/Nm, respectively, indicating the peeled off particles from the coating surface due to the local plastic deformation occurred during pin-on-disk technique, and thus these worn particles and oxides on the surface were the sign of weight gain on this sample and not weight loss.

(4) The minimum friction coefficient belonged to pure nickel sample by 0.915 and by B_4C participation in the Ni matrix, the friction coefficient was grown, and the lowest friction coefficient in all Ni-B₄C samples was for Ni-B₄C sample by 1.255 deposited at 10 A/dm² and the highest friction coefficient was to be 1.594 for the Ni-B₄C baseline sample.

(5) The morphology after wear of the samples indicated the adhesive wear as the prominent mechanism for pure nickel sample and to a high degree abrasive wear appeared to be the dominant in the Ni-B₄C nanocomposite samples. The reduction of adhesive wear signs in the Ni-B₄C samples was accompanied by the diminishment of wear rate, plastic deformation, ploughing, and delamination that definitely led to improve wear resistance, although by rising the pulse frequency from 10 to 100 Hz, the growth of adhesion, plastic deformation, and ploughing resulted into the increased wear rate compared to the of Ni-B₄C baseline sample.

(6) All in all, the optimum electrodeposition condition for Ni-B₄C nanocomposite coatings was obtained at 10 A/dm², $\gamma = 50\%$, and f = 10 Hz for a coating possessing the minimum weight loss (-0.00023 gr), the lowest wear rate (-0.552×10⁻⁴ mm³/Nm), the maximum incorporation rate (6.25 Vol% B₄C) among pure nickel and Ni-B₄C coatings, and the lowest friction coefficient (1.255) among all Ni-B₄C coatings. Finally, it was concluded that increasing current density and duty cycle improved the wear resistance while the enhancement pulse frequency led to worsen it.

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