Effect of plating parameters on microstructure and tribological properties of Co–BN(hexagonal) nano composite coatings

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Abstract: Effects of current density, duty cycle and frequency on microstructure and particles content of electrodeposited Co–BN (hexagonal) nano composite coatings were analyzed by SEM, FESEM, EDS, AFM and XRD techniques. The microhardness, tribological behavior and wear mechanism were also investigated. Generally, as the current density and frequency increased, the particles content and microhardness of the coatings increased firstly and then decreased. Moreover, by reducing duty cycle, more particles were incorporated and higher microhardness was obtained. The best tribological behavior was achieved under the conditions: duty cycle of 10%, frequency of 50 Hz and current density of 100 mA/cm².

Key words: Co–BN coating; frequency; duty cycle; electrodeposition; composite; microhardness; tribological behavior

1 Introduction

Pulse electrodeposition is one of the most technological methods for co-depositing fine (micro or nano) particles with a metal or alloy matrix to produce composite coatings for potential engineering applications [1−4]. The properties of metal matrix composite coatings produced by electroplating depend on many process parameters including bath composition, temperature, pH, current density, duty cycle, frequency and many variations [5,6]. So, in order to obtain composite coatings with high quality, it is necessary to control the electrodeposition parameters [7]. The purpose for incorporation of hard particles such as SiC, TiC, Al₂O₃, ZrO₂ into metal deposits is to increase the wear resistance and hardness of coatings. Enhanced lubricity has been achieved by incorporating PTFE, MoS₂ particles [5,8,9]. Recently, nanocrystalline Co and Co-based alloys have been identified as an alternative coating to hexavalent chrome plating in helicopter dynamic components, military aircraft (landing gear, hydraulic actuators, gas turbine engines and propeller hubs) and in decorative applications [10−12]. However, like many hard coatings, the surface of cobalt deposits includes a thin oxide film that increases during the tribological test and leads to a substantial friction coefficient and wear rate [10]. On the other hand, hexagonal boron nitride (BN(h)) particles with similar crystal structure to graphite is a solid lubricant that exhibits a very low friction coefficient and wear rate in composite deposits. It possesses a number of interesting properties such as high temperature stability, extreme pressure performance and inertness in a wide variety of chemical environments [13−15]. To the best of our knowledge, there are no reports on incorporating nano BN(h) particles into Co films. Therefore, the use of BN(h) particles appears attractive for cobalt matrix reinforcements. The novelty of the present approach relies on the facility of producing Co–BN(h) films by electrodeposition due to the necessity of dry coatings for modern technologies like aerospace, aircraft and automobile industries, and with the purpose to improve the properties of the produced coatings by controlling plating parameters. Accordingly, in this work the effect of pulse electrodeposition parameters and characterizations of Co–BN(h) nano composite coatings were investigated.

2 Experimental

Co–BN(h) nano composite coatings (and pure...
cobalt for comparison) were deposited on copper substrates from a chloride solution containing 210 g/L cobalt chloride, 20 g/L boric acid, 0.25 g/L saccharine and 5 g/L BN(h) particles (with diameter of less than 100 nm). The copper substrates with a dimension of 30 mm×30 mm were ground, then degreased in Uniclean 675 solution and chemically cleaned in acetone and distilled water for 4 min, then activated in chloride acid for 30 s. Prior to the electroplating process, the electrolyte was under ultrasonic waves for 30 min, and during the process it was in suspension in an agitating bath solution with the use of a 300 r/min magnetic stirrer placed at the cell bottom. The deposition time was 120 min. After each experiment, the pure cobalt plates (used as anode) were ground using a 60 grade silicon carbide paper. In pulse deposition experiments, the frequency (i.e. $f=\frac{1}{(t_{\text{on}}+t_{\text{off}})}$), duty cycle (i.e. $d=\frac{t_{\text{on}}}{(t_{\text{on}}+t_{\text{off}})}$) and current density of the imposed rectangular pulses were varied. The deposition parameters are listed in Table 1. The characteristics of the coatings were evaluated by scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM) equipped with an energy dispersive spectroscopy (EDS), atomic force microscopy (AFM, NanoScope II Version 5.12r2), X-ray diffraction (XRD, Philips XPert-Pro) with Cu Kα radiation. The hardness of the coatings was determined using a Vickers microhardness instrument at an applied load of 100 g for 10 s. Finally, the wear resistance and friction coefficient of the composite coatings were investigated using a ‘pin-on-disk’ tester at the constant sliding speed ($v=0.1$ m/s) and normal load ($W=2$ N). The pin was made of AISI 52100 steel with diameter of 5 mm.

<table>
<thead>
<tr>
<th>Table 1 Deposition parameters</th>
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<tr>
<td>Parameter</td>
</tr>
<tr>
<td>pH</td>
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<tr>
<td>Temperature/°C</td>
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<tr>
<td>Current density/(mA·cm⁻²)</td>
</tr>
<tr>
<td>Duty cycle/%</td>
</tr>
<tr>
<td>Pulse frequency/Hz</td>
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</table>

3 Results and discussion

3.1 Surface morphology of BN(h) coating

Figure 1 displays the SEM image, EDS elemental mapping and XRD pattern of the selected Co–BN(h) nano composite coatings with approximately 4.1% (mass fraction) BN(h). The thickness of as-deposited coatings is about 23 μm. Morphological analyses confirm the possibility of homogeneity, compactness and smoothness deposition by electroplating method. The micrograph and XRD pattern depicted in Fig. 1 are also typical of electrodeposited nano coatings obtained under these deposition parameters with the crystal size of 15 nm. Moreover, the EDS analysis reveals uniform distribution of the BN(h) particles throughout the matrix of cobalt that is electroplated by virtue of magnetic stirring, and no particle agglomerations can be distinguished within the coatings, which is an important factor to gain an appropriate composite coating [16].

![Fig. 1 Secondary SEM image (a), EDS analysis (b) and XRD pattern (c) of Co–BN(h) nano composite coating obtained at constant frequency, duty cycle and current density of 50 Hz, 10% and 100 mA/cm², respectively](image-url)
3.2 Effect of pulse current density

In order to study the effect of pulse current density on microstructural changes of Co−BN(h) nano composite coatings, detailed analyses of X-ray diffraction (XRD) patterns were carried out, as shown in Fig. 2. The diffraction peaks of the deposited coatings indicate the nanocrystalline character of the films. All deposits have hexagonal closed pack (HCP) structure (JCPDS No. 89-4308). It is a well established fact that the Co film favors an HCP structure in this range of current density [17,18]. The XRD patterns show main peaks corresponding to (100), (002) and (110) crystallographic planes at 2θ positions of 41.48°, 44.37° and 75.83°, respectively. Some weak diffraction lines from composite coatings at high current density are also present. Figure 2 shows that the intensity of (002) peak is the highest in most of the patterns, indicative of (002) preferred orientation. However, the composite coating which is deposited at 250 mA/cm² does not follow above trend, and exhibits a predominant (100) reflection accompanied with (110). This change in the preferred orientation can be also responsible for the lower microhardness of this composite as compared to the others. The crystallite size of coatings was calculated using the angular width of main peaks at full width half maximum in conjunction with Scherrer equation [19], as listed in Table 2.

It is obvious that the crystallite size of coatings has an increasing trend (from ~15 to 46 nm) by increasing the current density (from 100 to 250 mA/cm²). The crystallite size of the as-deposited coatings was also analyzed by FESEM and AFM with nano metric resolution, for further verifying our conclusions from the Scherrer equation. Figure 3 shows that the Co−BN(h) nano composite coating deposited at current density of 250 mA/cm² has small grains in order of 45−50 nm, which is in agreement with the results in Table 2. In general, it is expected that the crystallite sizes of the electrodeposited coatings decrease by increasing the current density, because an increase in the current density results in a higher overpotential that reduces the energy of nucleus process and hence increases the nucleation rate [20−22]. Nevertheless, several authors have reported an increase of deposit’s crystallite size with increasing the current density [7,23], which is similar to this research. This outcome has been attributed to the hydrogen codeposition at the cathode-electrolyte interface and a decrease of the deposition efficiency. In fact, the changes in the growth mechanisms and surface energy in the presence of hydrogen are responsible for the increased crystallite size of deposits.

Table 2 Variations of crystal size as a function of current density

<table>
<thead>
<tr>
<th>Current density/(mA·cm⁻²)</th>
<th>50</th>
<th>100</th>
<th>150</th>
<th>200</th>
<th>250</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crystallite size/nm</td>
<td>19±1</td>
<td>15±1</td>
<td>16±2</td>
<td>27±1</td>
<td>46±3</td>
</tr>
</tbody>
</table>

Fig. 2 XRD patterns of Co−BN(h) nano composite coating electrodeposited at different current densities: (a) 50 mA/cm²; (b) 100 mA/cm²; (c) 150 mA/cm²; (d) 200 mA/cm²; (e) 250 mA/cm²

Fig. 3 FESEM (a) and AFM (b) images of Co−BN(h) nano composite coating deposited at constant current density of 250 mA/cm²

The mass fraction of BN(h) particles and microhardness of the coatings as a function of current density are also investigated in Fig. 4. As it can be seen, the incorporation behavior initially shows a rapid increase in codeposition due to the increasing tendency for adsorbed particles to achieve the cathode surface. Totally, the particles/electrode interaction is in competition with particles removal from the cathodic
surface by the suspension hydrodynamic forces [24]. At low current densities (below 100 mA/cm\(^2\)), there is a weaker electric force amongst the particles and surface, which results in a lower fraction of the BN(h) particles in the coatings. Then by increasing pulse current density (up to 150 mA/cm\(^2\)), which leads to a faster deposition of metal matrix ions, the content of incorporated particles decreases. In addition, the decrease in BN(h) particles incorporation at this higher current density can be ascribed to the electrochemical potential, which affects the adsorption of particles on the substrate surface [25]. Further increase in current density from 150 to 250 mA/cm\(^2\) leads to a marginal increase in the electrodeposited particle content as a consequence of decreased cobalt deposition due to the cobalt’s depletion near the surface. It is evident from the Vickers microhardness values that at the initial part (in the range of 50–100 mA/cm\(^2\)), a high tendency of increasing hardness is appearing as the codeposition BN(h) particles is increased. However, beyond this current density, the microhardness begins to decline. As expected, the hardness decreases due to the lower incorporation of the particles in the cobalt film up to a 200 mA/cm\(^2\). However, the striking decrease at higher current density in the hardness of the Co composite coatings can be attributed to the change in crystallite size and preferred orientation, which was mentioned above. The changes of hardness are not only related to the amounts of particles incorporation [23,25]. The main factor governing microhardness of the composites in this range of current density is the structure of the metal matrix, which overlaps the particle hardening effect and in turn alters the coating hardness.

Fig. 4 Effect of current density on mass fraction of codeposited BN(h) particles and microhardness of electrodeposited films at constant frequency of 50 Hz and duty cycle of 10%.

On the other hand, tribological properties are another important factor governing the industrial application of surface coatings. Average friction coefficient and mass loss were calculated after a sliding distance of 300 m, subjected to dry sliding wear test (with average coating thickness of 25 μm). Figure 5 displays the changes in the friction coefficient and mass loss of as-plated coatings as a function of pulse current density. It can be found that the average friction coefficient drops with increasing current density up to 100 mA/cm\(^2\), due to the increased BN particles content, and then increases. It implies that an increase in the content of BN(h) particles in the deposit, as a lubricant, with uniform distribution greatly enhances the lubricating properties of the composite coating which is in accordance with Refs. [13,15]. With regards to the wear resistance, characterized by the mass loss, it is obvious that with the increase of current density from 50 to 200 mA/cm\(^2\), the mass loss of composite coatings decreases from 2.2 to 0.6 mg, and then it increases again with further increase of current density up to 250 mA/cm\(^2\). It can be seen that the wear resistance of the composite coatings is dependent on microhardness indicating that higher wear resistance occurs at higher hardness for composite coatings. Since wear is the result of plastic deformation accumulation and the measurement of a material’s ability to combat plastic deformation is hardness, the correlation between them has been comprehensively studied. The relation between the wear resistance and microhardness can be expressed by Archard’s equation:

\[ V = \frac{k W x}{H} \]

where \(V\) is the volume wear; \(x\) is the sliding distance; \(k\) is the non-dimensional wear coefficient; \(W\) is the normal load and \(H\) is the hardness of the surface being worn away [26,27]. According to this equation, there is an inverse correlation between the wear behavior and surface hardness, and the coating wear decreases as its surface hardness increases, which justifies the outcomes of this work.

Fig. 5 Variations of friction coefficient and mass loss of composite coatings as function of current density deposited at constant frequency of 50 Hz and duty cycle of 10%
After wear tests, the morphology of the coatings was also investigated. Figures 6(a) and (b) show the selected SEM images taken from the worn surfaces of composite coatings deposited at the current density of 50 and 250 mA/cm², respectively. With respect to wear appearance, the worn tracks on all the composite coatings have various scratches and grooves parallel to the moving direction of the pin, which are typical for abrasive wear [25]. Moreover, at higher current densities (more than 50 mA/cm²), the presence of torn patches and some detachment within the worn tracks is typical evidence for plastic deformation and thus the adhesive wear. Changing of current density due to the change of crystal size and microhardness causes a change in wear mechanism as predominant abrasion to the mixed abrasive and adhesive wear. With increasing current density up to 250 mA/cm², this characteristic becomes more obvious and deeper grooves are observed on the worn surface (Fig. 6(b)). The dominant mechanism of adhesion at this current density together with deeper grooves can be attributed to predominant (100) crystallographic orientation and higher crystallite size.

3.3 Effect of duty cycle

The effect of duty cycle on the microstructural changes of deposits is shown in Fig. 7. The XRD diagrams of deposits prepared at low duty cycles reveal preferred orientation of (002) and (100) planes, but an increase in duty cycle results in the worsening of the (002) texture and leads to a change in the preferred orientation of the deposits from predominant (002) to the (100) and (110) random orientations. According to crystallite size values in Table 3, it is clear that the change of duty cycle strongly affects the crystallite size of the deposits. The crystallite size of the deposit at duty cycle of 10% is found to be about 15 nm, increasing duty cycle up to 70% results in coarse grains of 53 nm. At lower duty cycle due to a minus t off, less time is required for grain growth and thus, finer grain structure can be obtained.

![Fig. 6 SEM images of worn surfaces of composite coatings deposited at current density of 50 mA/cm² (a) and 250 mA/cm² (b)](image)

![Fig. 7 XRD patterns of Co–BN(h) nano composite coating electrodeposited at different duty cycles: (a) 10%; (b) 30%; (c) 50%; (d) 70%](image)

<table>
<thead>
<tr>
<th>Duty cycle/%</th>
<th>10</th>
<th>30</th>
<th>50</th>
<th>70</th>
</tr>
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<tbody>
<tr>
<td>Crystallite size/nm</td>
<td>15±1</td>
<td>24±3</td>
<td>39±2</td>
<td>53±3</td>
</tr>
</tbody>
</table>

The variation of the amount of BN(h) particles incorporated and microhardness of the deposits as a function of duty cycle are shown in Fig. 8. The mass fraction of the incorporated BN(h) particles in the composite coatings decreases by increasing the duty cycle and the highest particle incorporation (4.116%) is observed at the lowest duty cycle (10%). According to Refs. [28,29], effects of duty cycle and pulse frequency on the particles content can be characterized by introducing of t on and t off (the time at which no current passes through the system). Taking into account that decreasing the duty cycle corresponds to an increase of t off, more BN(h) particles as compared to the metal ions would be deposited more strongly on the cathode surface, and as a result the mass fraction of the incorporated...
particles would increase at lower duty cycle. It also demonstrates a dependence of the microhardness values of the composite deposits on the duty cycle imposed. From Fig. 8, it can be seen that the hardness of the composite coating is mainly related to the amount of BN(h) particle incorporation. This behavior can be related to the dispersion strengthening, because the particles impede the motion of dislocations and act as barriers to retard the plastic deformation of Co matrix and hence increases the microhardness [23].

The results discussed above represent that plating parameters significantly influence the particles content and the hardness of the as-deposited coatings. Thus, for more investigation, the effect of duty cycle on friction coefficient and wear resistance was also studied. Figure 9 shows the average friction coefficient of Co–BN(h) nano composite coatings obtained at different duty cycles of 10%, 30%, 50% and 70%. It shows that the composite coatings produced at lower duty cycle have a lower friction coefficient than those obtained at higher duty cycle. The friction coefficient increases from a low value of 0.34 to about 0.79 with the increased duty cycle from 10% to 70%. By comparing Fig. 9 to Fig. 8, it can be concluded that higher BN(h) content brings minor friction coefficients. Figure 9 also shows the variation of mass loss of composite coatings with duty cycle. By increasing the duty cycle, the mass loss value of the composite coatings first increases and then decreases, revealing their better wear resistance at lower duty cycles. It demonstrates that the hardness of the electrodeposited composite coating has the most significant effect on the wear behavior of the coating. In other words, the results are in agreement with Archard law [26].
deformations along the sliding directions on the worn surfaces of the coatings deposited at higher duty cycles of 50% and 70% indicate that adhesive wear also takes place. This change in wear mechanism is caused by crystallite size effects as well as changing of crystallographic orientation from (002) to (100).

3.4 Effect of frequency

Figure 11 shows the results of the XRD analysis of Co–BN(h) composite electrodeposited at different frequency. The coatings that are orientated in the (100), (002) and (110) plane directions, represent hexagonal closed pack (HCP) structure, with the highest reflection intensity corresponding to the (002) preferential crystallographic orientation at moderate frequencies (10–50 Hz) and the highest reflection of (100) at low frequency (1 Hz). However, the XRD pattern of the deposited coating at high frequency of 100 Hz shows face centered cubic (FCC) structure by the presence of three lines of (111), (200) and (311), which distinguishes the two cobalt phases from each other [10]. Thus, the crystalline orientation and microstructure of the deposits can be modified by controlling the deposition parameters. The observed transition in crystal structure may be ascribed to the overpotential, thickness of the diffusion layer or hydrogen adsorption/evolution, and other adsorption processes. Although, it is difficult to determine how these variables separately or in combination cause the observed structural transformation to the FCC phase at high frequency of 100 Hz. In addition, changing of frequency has an effect on the crystallite size of deposits. As shown in Table 4, the driving force of nucleation increases with increasing frequency up to 50 Hz, while causing grain refinements (i.e. 15 nm). But further increasing of frequency doesn’t have significant effect on the value of crystallite size which remains almost constant. The increased nucleation rate at moderate frequencies is attributed to the enhancement of migration of ions owing to the thin pulse diffusion layers [30]. As listed in Tables 3 and 4, the variation of crystallite size as a function of duty cycle is sharper and thus duty cycle has a more pronounced effect on the crystallite size of coatings than pulse frequency. It represents that the ratio of \( t_{\text{off}} \) to \( t_{\text{on}} \) (exposed by duty cycle) is more influential on the crystallite size than the length of \( t_{\text{off}} \) (indicated by frequency).

The effects of frequency on the particles content and microhardness of corresponding films are shown in Fig. 12. It shows that BN(h) particle incorporation tends to increase with changes in the pulse frequency from 1 to 50 Hz, while further increase of the frequency will decrease the incorporated particles in the composite coatings in turn. The results obtained by LAJEVARIDI and SHAHRABI [19] showed a decrease in particles content of the coatings, which is at range beyond 10 Hz for Ni–TiO₂ nano composite coatings. In fact, at low frequencies (lower than 50 Hz in this work), BN(h) particles, which are loosely adsorbed on the surface, can be removed by the presence of hydrodynamic forces. Moreover, the higher reduction rate of free metal ions in contrast with ions adsorbed on BN(h) particles, due to the depletion of electrolyte near the cathodic surface, reduces the particles content of the coatings. By increasing the frequency up to 50 Hz, less loosely adsorbed particles can be intercepted. However, the effects of capacitance and consequently incomplete discharge of ions adsorbed on particles cause a second decrease in the BN(h) particles content of deposits at higher frequencies (beyond 50 Hz). Furthermore, the concentration gradient of BN(h) particles near the surface cannot be removed in the short \( t_{\text{off}} \) and

Table 4 Variations of crystallite size as function of frequency

<table>
<thead>
<tr>
<th>Frequency/Hz</th>
<th>Crystallite size/nm</th>
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<tbody>
<tr>
<td>1</td>
<td>31±2</td>
</tr>
<tr>
<td>10</td>
<td>24±2</td>
</tr>
<tr>
<td>50</td>
<td>15±1</td>
</tr>
<tr>
<td>100</td>
<td>15±1</td>
</tr>
</tbody>
</table>

Fig. 11 XRD patterns of Co–BN(h) nano composite coating electrodeposited at different frequency: (a) 1 Hz; (b) 10 Hz; (c) 50 Hz; (d) 100 Hz

Fig. 12 Effect of frequency on mass fraction of codeposition BN(h) particles and microhardness of electrodeposited films at constant current density of 100 mA/cm² and duty cycle of 10%
accordingly leads to lower particle content in the coatings. As shown in Fig. 12, the increase of microhardness corresponds to an increased particle content in the deposits, and the maximum microhardness (HV441.26) as well as maximum BN(h) incorporation is seen at a frequency of 50 Hz. These results are also in consistent with the effect of frequency on the crystallite size of the coatings. To conclude, higher microhardness of the composite coatings is due to a combination of the particles reinforcement and the change of cobalt structure. Therefore, it seems that there is an optimum cycles of current pulses (proper combination of $t_{on}$ and $t_{off}$) determined by the imposed frequency, which permits a sufficient incorporation of particles in the cobalt matrix and harder coatings [30,31].

Figure 13 shows the friction coefficient and mass loss versus frequency of electrodepositing Co-BN(h) nano composite coatings at constant duty cycle (10%) and current density (100 mA/cm$^2$). The pin-on-disc results indicate that, with increasing frequency, the friction coefficient decreases from 0.72 to 0.34, and then increases to 0.57. This could be correlated to the observed incorporation content of particles by variation of the duty cycles. The relatively higher amount of BN(h) particles produced at frequency of 50 Hz (4.1%) results in a decrease of friction coefficient of the coating. Moreover, the results of mass loss show that the wear resistance increases firstly and then decreases as frequency increases, which is consistent with the microhardness results as shown in Fig. 12.

![Fig. 13 Variations of friction coefficient and mass loss of composite coatings as function frequency deposited at constant current density of 100 mA/cm$^2$ and duty cycle of 10%](image)

4 Conclusions

Co–BN(h) nano composite coatings were produced by pulse electrodeposition. The effects of current density, duty cycle and frequency on microstructure, particles content, microhardness and tribological behavior of the coatings were investigated. The results showed the dependence of coating properties on the plating parameters as follows.

1) The crystallite size of the coatings increased with increasing duty cycle and current density (>100 mA/cm$^2$), but decreased with the increase of pulse frequency.

2) An increase of current density or duty cycle and a decrease of frequency led to a change in the preferred
orientation from the (002) to (100). Increasing frequency (100 Hz) led to change in coating structure from HCP to FCC.

3) In general, the particles content and microhardness increased initially and then decreased, when the current density and frequency increased. Decreasing the duty cycle from 70% to 10% resulted in an increase in particles content and microhardness.

4) The coatings deposited on the conditions: duty cycle of 10%, frequency of 50 Hz and current density of 100 mA/cm², exhibited an improvement in friction coefficient and wear resistance, due to the enhanced 

5) Generally, two wear mechanisms were present including abrasive wear and adhesive wear in these types of coatings at different plating conditions.

References


电沉积参数对 Co–BN(六方)纳米复合涂层显微组织和摩擦性能的影响

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University of Tehran, Tehran, Iran

摘要：通过扫描电镜、场发射扫描电镜、能谱、原子力显微镜和 X 射线衍射等测试技术，研究电流密度、占空比和频率对电沉积 Co–BN(六方)纳米复合涂层的显微组织和 BN 粒子含量的影响。同时，研究其显微硬度、摩擦性能以及磨损机理。随着电流密度和频率的增加，涂层的 BN 粒子含量和显微硬度先增加后减少。另外，通过减少占空比，可以沉积更多的 BN 粒子并且得到更高的显微硬度。在占空比 10%，频率 50 Hz 和电流密度 100 mA/cm² 的条件下，可以获得最佳的涂层摩擦性能。

关键词：Co–BN 涂层；频率；占空比；电沉积；复合材料；显微硬度；摩擦行为

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