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Preparation and Properties of Nanocomposite Coatings by Pulsed Current-Jet Electrodeposition

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To improve the surface quality and properties of nanocomposite coatings, a method using pulsed current-jet electrodeposition was adopted to prepare Ni-Al₂O₃ nanocomposite coatings. The effects of nanoparticle concentration and pulse current on the surface morphology, grain size and performance of the coatings were examined. The surface morphology of the coatings and the distribution of nanoparticles were analyzed by scanning electron microscopy. The microstructure and grain size of the coatings were studied by X-ray diffractometer. The microhardness and corrosion resistance of the coatings were characterized by microhardness tester and electrochemical workstation, respectively. The results showed that, adding proper amount of nano-Al₂O₃ in plating solution can improve the microhardness and corrosion resistance of the coatings. The coatings prepared by pulsed current-jet electrodeposition possessed smoother surface morphology, finer grain size and better dispersion of nanoparticles compared with the coatings prepared by direct current-jet electrodeposition. The former exhibited higher microhardness and better corrosion resistance compared with the latter.

Keywords: jet electrodeposition; pulse current; nanocomposite coatings; microhardness; corrosion resistance

1. INTRODUCTION

The failure from metal part corrosion has become the main reason for the short service life of wind power equipment. The usual anticorrosion measure is to cover material surface with protective coatings [1]. Nanocomposite coatings formed by co-deposition of nanoparticles and matrix metals are favored because of their excellent protective performance [2-5]. As the second phase, nanoparticles can not only transfer their properties to the coatings, but also provide a large number of nucleation sites for the deposition process [6-7]. Serkan Özkan has prepared Ni-SiC nanocomposite coatings by

electrodeposition, which improved coating corrosion and wear resistances [8]. A. C. Ciubotariu has obtained Ni-Al₂O₃ composite coatings under mechanical agitation, which improved coating corrosion resistance [9]. Yujun Xue has obtained Ni-CeO₂ nanocomposite coatings with ultrasonic irradiation. The microhardness and wear resistance of the resulting coatings were found to be increased [10].

Although Ni-Al₂O₃ nanocomposite coating possesses excellent performance owing to Al₂O₃ particles with high hardness, great thermal stability and good chemical inertness [11], the surface effect of nanoparticles allows them to agglomerate extremely easily [12]. The agglomerated particles increase the coating defects and decrease the coating properties. Furthermore, The average current density used in conventional electrodeposition technology is generally below 10 A/dm² [13-17]. The low current density reduces nucleation rate, and thus the surface of coatings forms cellular bulges to affect the surface quality [18-19]. In order to improve the surface quality and properties of composite coatings, PC-jet electrodeposition was used to prepare Ni-Al₂O₃ nanocomposite coatings in this study. The impact of jet-flow and the introduction of pulsed power supply can greatly improve the limiting current density. The higher peak current density (160 A/dm²) which was used in this experiment increased the nucleation rate and achieved the purpose of grain refinement. In addition, the nanoparticles and nickel ions were effectively transported to the cathode surface during the pulse off-time (T_{off}), so as to increase the dispersion of nanoparticles and improve the surface quality of composite coatings.

2. EXPERIMENTAL SYSTEM

2.1. Composition of plating solution and process parameters

The composition of plating solution is shown in Table 1. $NiSO_4$ is the main source of nickel ions in the solution. $NiCl_2$ is an anodic activator to prevent anodic passivation. H_3BO_4 works as a buffer reagent to maintain plating solution pH. Saccharin was added as a brightener to reduce stress and refine grain size.

Composition	Amount (g/L)	pН	Purpose	
NiSO ₄ ·6H ₂ O	280		Ni ²⁺ source	
NiCl ₂ ·6H ₂ O	40	4 + 0 1	Anodic activator	
H_3BO_4	40	4 ± 0.1	Buffering	
Saccharin	5		Reduce stress	
Al_2O_3	0, 4, 8, 12, 16, 20, 24		Second phase	

 Table 1. Composition of plating solution

The nanoparticles added to the plating solution in this experiment are $alpha-Al_2O_3$ with a size of 30 nm (Fig. 1). First, the desired amount of nanoparticles were suspended in deionized water by

ultrasonic oscillation for 60 min and then poured into the plating solution for ultrasonic oscillation for an additional 30 min. Due to its good conductivity and convenient pretreatment, the graphite sheets (30 mm \times 20 mm \times 3 mm) were used as substrates. Prior to experiments, the sheets were pretreated, which included sanding, degreasing and cleaning. In order to investigate the effects of the concentration of nanoparticles and the pulse current on the properties of the coatings, the concentration of nanoparticles was taken as the variable. The comparison experiments were performed by DC-jet electrodeposition and PC-jet electrodeposition. The process parameters are shown in Table 2.



Figure 1. SEM image of nano-Al₂O₃

Table 2. Process	parameters
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Parameter	Value	
Rate of flow/(L/h)	200	
Nozzle exit section size/mm ²	1×20	
Anode-cathode distance/mm	2	
Movement speed/(mm/s)	9	
Average current density/(A/dm ²)	80	
Duty ratio/%	50	
Pulse frequency/Hz	5000	
Temperature/°C	50 ± 1	
Electrodeposition time/min	60	

2.2. Structural characterization and performance test

The surface morphology of the resulting coating was characterized using a scanning electron microscopy (S-4800; Hitachi Instruments, Inc., Tokyo, Japan). The composition of the coating was analyzed by energy spectrometer attached to the scanning electron microscopy. The internal structure of the coating was analyzed by X-ray diffractometer (DMAX-2500PC; Rigaku Corp., Tokyo, Japan) under the conditions of 20-100° diffraction angles, CuK_a radiation source, 0.15406 nm wavelength, 40 kV, 150 mA, 2° step width, and 10°/min scanning speed. Potentiodynamic polarization curves were

generated on an electrochemical workstation (CHI660E; Chenhua Instruments Co., Shanghai) to evaluate the corrosion resistance of the coating. Microhardness was detected by vivtorinox microhardness tester (HXS-1000AK; Bsida Instruments Co., Chengdu). The effects of substrate materials on the resulting coating microhardness were eliminated by setting the applied load at 50 g and the load duration at 10 s. The microhardness of each workpiece was measured at 5 different positions and the average value was calculated to evaluate the microhardness of the coating.

3. RESULTS AND DISCUSSION

3.1. Surface morphology



Figure 2. Surface micrographs of the coatings prepared by DC-jet electrodeposition: (a) 0 g/L, (b) 8 g/L, (c) 16 g/L, (d) 24 g/L

The surface micrographs of the coatings prepared by DC-jet electrodeposition are shown in Fig. 2. The surface of pure nickel coating (Fig. 2a) formed cellular bulges. While the surface of composite coatings (Fig. 2b, c, d) was relatively smooth, with visible white particles. Such white particles were also observed in the preparation of Ni-Al₂O₃ composite coatings by Aruna [20]. The higher the concentration of nanoparticles, the more white particles. When the concentration was too high, the white particles agglomerated. As the concentration of nanoparticles increased, surface defects became more apparent, such as micropores and pits. The composition of composite coating was analyzed using an energy spectrometer. According to the resulting energy spectra (Fig. 3), the oxygen

and aluminum content in the white particles was very high, whereas the gray area only contained nickel. Therefore, it can be inferred that the white particles are Al_2O_3 . According to reports [21], Y. Sun has prepared Ni- Al_2O_3 composite coatings, and there were many cellular bulges on the surface of the coating because the maximum current density was only 3.4 A/dm².



Figure 3. Cross-sectional image of Ni-Al₂O₃ nanocomposite coating (a) and its energy spectra (b)

In the process of jet electrodeposition, the electric field intensity of the convex regions of the coating surface is higher because of the influence of the "effect of point" (Fig. 4a). The cellular bulges became larger and larger because the nickel ions were preferentially deposited in the convex regions. When appropriate amount of nanoparticles were added to the plating solution, the nanoparticles were adsorbed onto the concave regions of the coating surface under the impact of jet-flow. These concave regions were indirectly filled by nanoparticles, which made the distribution of electric field lines more uniform (Fig. 4b). As a result, the surface of composite coating was smoother than that of pure nickel coating. However, when the nanoparticle concentration was too high, agglomeration was facilitated because of the increase of particle collision probability [22]. The agglomerated particles embedded in the composite coating decreased the homogeneity of current distribution [23]. Furthermore, large numbers of nanoparticles in the plating solution occupied a lot of volume, leading to a serious shortage of nickel ions around the cathode.



Figure 4. Sketch map of coating growth by DC-jet electrodeposition: (a) Pure nickel coating, (b) Composite coating

Hydrogen was precipitated rapidly because there was not enough nickel ions to participate in the reduction reaction. The impurities were generated in the hydrogen evolution reaction on the coating surface, resulting in the increase of coating defects.

The surface micrographs of the coatings prepared by PC-jet electrodeposition are shown in Fig. 5. The surface of pure nickel coating (Fig. 5a) was smooth and had no obvious cellular bulges. The surface defects of composite coatings (Fig. 5b, c, d) were obviously reduced, and the distribution of nanoparticles was relatively uniform. Lajevardi had prepared Ni-Al₂O₃ nanocomposite coating under ultrasonic agitation. The nanoparticles were uniformly distributed in the nickel matrix while the cellular bulges of the resulting coating surface were very obvious [24]. Other researchers also obtained similar results [25-26]. The main reason is that the current density used in traditional electrodeposition is very low. The schematic diagram of PC-jet electrodeposition is shown in Fig. 6. According to the two-step adsorption theory of Guglielmi [27], particles with adsorbed ions are initially weakly adsorbed on cathode surface, and then these particles are strongly adsorbed to the surface with the reduction of the adsorbed ions. The electric double layer was formed effectively around the cathode during the pulse off-time (T_{off}), and the particles with adsorbed ions were allowed to penetrate into the cathode surface better.



Figure 5. Surface micrographs of the coatings prepared by PC-jet electrodeposition: (a) 0 g/L, (b) 8 g/L, (c) 16 g/L, (d) 24 g/L

The peak current density during the pulse on-time (T_{on}) is twice as high as average current density because the duty ratio is 50%. Because of a higher peak current density (160 A/dm²), the distribution of electric field lines on the cathode surface was more intensive (Fig. 7), which improved the dispersion of the nanoparticles. Effective particle dispersion in the plating solution created more

opportunities for loose particle adsorption onto the electrode [28]. Furthermore, the impact of jet-flow can play a certain role in the agitation of the plating solution. Therefore, the adverse effects of nanoparticle agglomeration on composite coatings were weakened, and thereby avoiding the formation of voids and pits. Although the particle agglomeration was reduced by ultrasound assistance according to Qu's report, the use of ultrasonic vibration has resulted in reducing the amount of alumina embedded in composite coatings [29].



Figure 6. Schematic diagram of PC-jet electrodeposition



Figure 7. Sketch map of composite coating growth by PC-jet electrodeposition

The nano-Al₂O₃ content of the resulting coatings at different concentrations is shown in Fig. 8. The nano-Al₂O₃ content gradually increased with the increase of nanoparticle concentration. When the nanoparticle concentration held constant, the nano-Al₂O₃ content in the composite coatings prepared by PC-jet electrodeposition was higher than that in the composite coatings prepared by DC-jet electrodeposition. Previous study has shown that the particle content in the coatings prepared by PC electrodeposition was higher than that in the coatings prepared by DC electrodeposition [30]. This result was mainly due to the fact that more nanoparticles were transported to the cathode and adsorbed on its surface during T_{off} after the introduction of pulse power supply, increasing the probability that nanoparticles were embedded in the coating.



Figure 8. Nano-Al₂O₃ content (at.%) of the coating under different nanoparticle concentrations

3.2. Coating microstructure

The X-ray diffraction patterns of the coatings prepared by DC-jet electrodeposition and PC-jet electrodeposition are shown in Fig. 9. The crystal planes of the four diffraction peaks are (111), (200), (220), and (311). So the structure of coatings is face-centered cubic. The diffraction peak of nano- Al_2O_3 in the X-ray diffraction patterns of the composite coatings was not found. A possible reason is that the content of nanoparticles in the coatings is too low to be detected. When the concentration of nanoparticles was 16 g/L, the content of nanoparticles was only 1.54 at.% (Fig. 8). The preferred orientation of coatings is in the (111) crystal plane.



Figure 9. X-ray diffraction patterns of the coatings: (a) Direct current, (b) Pulse current

The preferred orientation in the (111) crystal plane was weakened by PC-jet electrodeposition. Moreover, the diffraction peaks of coatings were significantly wider than that of the coatings prepared by DC-jet electrodeposition. Wu concluded that the peak width of the automatically plated Ni-Al₂O₃ composite coating was broader than that of the manually plated composite coating [31]. Li also found that the peak width at half-maximum intensity of composite coating was broader than that of pure nickel coating, and the average grain size of composite coating was 16 nm while the average grain size

of pure nickel coating was 21 nm [32]. The grain size of each coating was analyzed using Scherrer equation [33]. It can be concluded that the grain sizes of the coatings prepared by DC-jet electrodeposition were 12.9, 12.7 and 13.5 nm when the concentration was 0, 8 and 16 g/L, respectively. While the grain sizes of the coatings prepared by PC-jet electrodeposition were 11.4, 11.1 and 12.4 nm. The grain size was reduced after the pulse power supply was introduced. According to reports [34], Ilaria Corni has prepared Ni-Al₂O₃ composite coatings, and the minimum grain size of the coating was up to 29 nm.

According to the theory of electrodeposition, the grain size is mainly determined by the nucleation rate and the grain growth rate. The faster the nucleation rate, the slower the grain growth rate and the smaller the grain size. When nanoparticles were added to the plating solution, the nanoparticles adsorbed onto cathode surface provided a large number of nucleation sites for the deposition process [35]. Moreover, these particles distributed at the crystal boundaries of matrix metal impeded the growth of grain, thereby reducing the grain size. After the introduction of pulsed power supply, the high peak current density raised the cathodic overpotential. According to the theory of crystallization, the higher the cathodic overpotential, the greater the nucleation probability, which made the microstructure of the coatings finer. In earlier reports [36], it has been reported that Al_2O_3 nanoparticles tended to agglomerate and formed larger particles with sizes bigger than 1 μ m when the particle concentration was too high. These agglomerated particles caused the coating surface to be uneven and reduced the nucleation sites, resulting in larger grain size.

3.3. Microhardness



Figure 10. Microhardness (HV) of the coating under different nanoparticle concentrations

The relationship between coating microhardness and nanoparticle concentration is shown in Fig. 10. With the increase of nano-Al₂O₃ concentration, the microhardness of coating prepared by DC-jet electrodeposition reached 536 HV. While the microhardness of coating prepared by PC-jet electrodeposition reached 562 HV, which was 19.6% higher than that of pure nickel coating prepared by DC-jet electrodeposition. These microhardness values are higher than that reported for Ni-Co/SiO₂ and Ni/nano-Al₂O₃ composite coatings [37-38]. However, the microhardness decreased slightly when

the concentration was too high. When the concentration held constant, the microhardness of coating prepared by PC-jet electrodeposition was higher than that of coating prepared by DC-jet electrodeposition. It has been reported previously that Chen prepared Ni-Al₂O₃ composite coatings by pulse electrodeposition, and the maximum hardness of the resulting coating was 520 HV [39].

Nano-Al₂O₃ particles can transfer their properties to the resulting coatings effectively. When these particles were dispersed in the coating as the second phase, they played the role of dispersion and dislocation strengthening. The X-ray diffraction patterns of the coatings (Fig. 9) showed that the grain size can be refined by adding proper amount of nanoparticles. According to the relationship of Hall-Petch, the microhardness of polycrystalline materials increases as grain size decreases [40]. Consequently, the effect of fine-grain strengthening was achieved. Góral found that the addition of nanoparticles can improve the microhardness of the Ni coatings, and a composite coating with microhardness of 418 HV was obtained [41]. After the introduction of pulsed power supply, the grain size was further refined, and thus the strengthening effect was enhanced. Furthermore, the nanoparticles near the cathode were replenished during T_{off} and there are more particles adsorbed on the cathode surface, thereby improving the content and dispersion of nanoparticles. The coating microhardness in the present case was further improved. In previous studies, Badarulzaman has prepared nickel-alumina composite coating with the aid of mechanical agitation and increased the content of particles, thus the coating microhardness was improved. Since the size of the particles added was 5 µm, the highest microhardness of the resulting coatings only reached 401.8 HV [42]. However, when the nanoparticle concentration was too high, the coating nanoparticle content tended to be stable because the particles adsorbed on the cathode surface were limited. Besides, the grain size became larger due to the agglomeration phenomenon, and thus the microhardness decreased.

3.4. Corrosion resistance

The potentiodynamic polarization curves of the coatings prepared by DC-jet electrodeposition and PC-jet electrodeposition in 3.5 wt.% NaCl solution are shown in Fig. 11. The corrosion potential (E_{corr}) and corrosion current density (i_{corr}) of coatings were obtained from the intersection of the cathodic and anodic Tafel curves using the Tafel extra polation method. The results are shown in Table 3. The corrosion potentials of the coatings prepared by PC-jet electrodeposition were more positive than that of the coatings prepared by DC-jet electrodeposition under the same concentration. The former exhibited smaller corrosion current density compared with the latter, which implied that the former possessed better corrosion resistance. By comparing PC deposition with DC deposition, Chang came to a similar conclusion [43]. With the increase of nano-Al₂O₃ concentration, the corrosion resistance of composite coatings increased rapidly and then decreased gradually. When the concentration of nanoparticles was 12 g/L, the corrosion resistance was optimal. It has been reported previously that A. Góral obtained the cortosion current density of Ni-Al₂O₃ composite coatings at different current densities, and concluded that the minimum value of i_{corr} was 4.37 μ A·cm⁻² [44].



Figure 11. Potentiodynamic polarization curves of the coatings in 3.5 wt.% NaCl solution: (a) Direct current, (b) Pulse current

Sam	ples	AlcOc	Al-O- E _{corr} /V		$i_{corr}/\mu A \cdot cm^{-2}$	
Direct	Pulse	(g/L)	Direct	Pulse	Direct	Pulse
current	current		current	current	current	current
а	a'	0	-0.271	-0.267	2.469	2.232
b	b'	4	-0.275	-0.261	2.430	2.126
с	c'	8	-0.269	-0.255	2.157	1.779
d	d'	12	-0.253	-0.245	1.796	1.191
e	e'	16	-0.259	-0.257	2.372	1.980
f	f	20	-0.289	-0.274	2.350	2.345
g	g'	24	-0.303	-0.282	2.849	3.025

Table 3. Corrosion potential and corrosion current density

As can be seen from Table 3, adding proper amount of nano-Al₂O₃ can improve the corrosion resistance of the coating. This can be attributed to finer grain size with the addition of nanoparticle and validated the previous findings [45-46]. The more compact coating made it difficult for the corrosive medium to penetrate into the coating. Furthermore, the chemical inertness of Al₂O₃ particles is good [47]. When these particles were dispersed in the coating as the second phase, they played a shielding role in the penetration of corrosive medium. Szczygie†also studied the corrosion resistance of Ni-Al₂O₃ composite coatings. However, the diameter of the particles added was up to several hundred nanometers, so the protective properties of the obtained composite coatings were difficult to be greatly improved [48]. After the introduction of pulsed power supply, more nickel ions were transported to the cathode during T_{off} due to the impact of jet-flow. The nanoparticles near the cathode were completely encapsulated by these nickel ions. When these nickel ions were reduced, these nanoparticles can be tightly embedded into the coating. Moreover, the impact of the jet-flow played a certain stirring effect on the plating solution. Hence the effects of agglomerated nanoparticles on corrosion resistance were significantly weakened. Du analyzed the coating microstructure and the results showed that the microstructure of the composite coating was much finer and denser compared with the pure nickel

coating [49]. In addition, the cathodic overpotential was increased owing to the higher peak current density, which reduced the critical nucleus radius of nucleation and avoided the formation of coarse grain. As a result, the smoothness and compactness of the coating can be improved and the corrosive chemicals were difficult to remain inside the coating. In other words, coating corrosion resistance was further enhanced. When the nano- Al_2O_3 concentration was too large, the relative lack of nickel ions around the cathode led to the intensification of the hydrogen evolution reaction. The impurities generated in the reaction were introduced into the coating, increasing coating defects. Corrosive chemicals easily penetrated into the internal space of coating through these defects, accelerating the corrosion rate and decreasing corrosion resistance.

4. CONCLUSION

After the introduction of pulsed power supply, the grain size was refined, and the dispersion of nanoparticles was improved. Moreover, the effects of agglomerated nanoparticles on composite coatings were weakened. Therefore, the surface of the coatings was smoother and more compact.

With the increase of nanoparticle concentration, the microhardness of the resulting coatings increased firstly and then decreased slightly. When the concentration remained constant, the microhardness of the coatings prepared by PC-jet electrodeposition was higher than that of the coatings prepared by DC-jet electrodeposition.

 $Ni-Al_2O_3$ nanocomposite coatings prepared by PC-jet electrodeposition exhibited better corrosion resistance compared with that prepared by DC-jet electrodeposition. When Al_2O_3 particles were at 12 g/L, the composite coating corrosion resistance was the highest. However, when the concentration was too high, the corrosion resistance decreased.

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