

EFFECT OF MATRIX ROUGHNESS ON MICROSTRUCTURE AND PROPERTIES OF ELECTROLESS Ni - P PLATING

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45 steel was chosen as the substrate to study the effect of matrix roughness on microstructure and properties of Ni-P composite coating in this paper. The morphology of the coating was observed by Scanning Electron Microscope. The microstructure was analyzed by X-ray diffractometer. The hardness, coat adhesion and wear resistance of the coating were tested respectively by microhardness meter, scratch spectrometer, friction and wear spectrometer. The results show that the coating phase structure is mainly amorphous Ni-P. The coating will be denser if the roughness is smaller, when the size of the particles on the coating surface is moderate and the distribution is uniform. When the surface roughness of the matrix is $0.0935\mu\text{m}$, the properties of Ni-P coating is the best, the microhardness of Ni-P coating is up to 1285.38HV, the bonding strength is up to 32.9N, the coating wear resistance is 0.0023mm^3 and the surface friction coefficient is 0.235.

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1. Introduction

The electroless plating is obtained after deposition by reducing metal ions in the bath into metal simple substance under the action of suitable reducing agent. It has been widely used in mechanical, aerospace, chemical, metallurgical and many other fields [1-3] because electroless plating can improve the hardness, wear resistance and corrosion resistance of the coating [4-7]. Pretreatment is one key process before electroless plating, including mechanical grinding, chemical pickling and activation and so on. The surface quality of electroless plating is different under different substrate pretreatment conditions, which affect the service life of the coating. Grinding is the first step of pretreatment, which determines the roughness of the substrate which will directly affect the quality of the coating. In a word, electroless plating with different pretreatment conditions will result in different surface conditions, which will affect the quality and service life of the coating [8-10].

There are numerous researches on acid pickling [11] and activation [12], while the study of substrate roughness on the quality and properties of electroless plating is relatively rare. The

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roughness of the substrate directly affects the smoothness of the substrate surface, thus affecting the quality of the coating.

Zhang [13] investigated the effect of substrate surface roughness on corrosion resistance of electroless nickel coating, and found that the lower the roughness, the better the corrosion resistance. Hua [14] proved that the number of active nickel atoms increases first and then decreases with the decrease of the surface roughness of the copper matrix. Liu [15] found that the surface roughness of the substrate has a certain influence on the uniformity of the coating thickness, and the higher the roughness is, the greater the thickness changes. However, the effect of substrate roughness on the microstructure, hardness, adhesion and wear resistance of Ni-P electroless composite coating was relatively little.

Therefore, 45 steel was chosen as the substrate to study the effect of matrix roughness on the microstructure, hardness, adhesion and wear resistance of Ni-P composite coating in this paper.

2. Experiment

2.1 Sample preparation

The experimental object is a 20mm×20mm×5mm 45 steel matrix. The technological process is as follows. Firstly, the steel matrix was polished with the sand paper of 140[#], 320[#], 500[#], 1200[#], hence the different roughness. The roughness Ra of matrix is measured to be 0.9635, 0.6750, 0.1558 and 0.0935μm (No. 1-4), respectively, by LEXT OLS5000 confocal microscope. Secondly, the sample was deoiled, rust removed, electroactivated, cleaned by deionized water, blown dry and then put into the plating bath. The formula of activation solution is shown in the following Table (1), pH was adjusted to 4 or so. Plating bath was formulated(30g/L NiSO₄·6H₂O, 25g/L NaH₂PO₂·H₂O, 15g/L Na₃C₆H₅O₇·2H₂O, 35g/L CH₃COONa, 25mL/L C₃H₆O₃, 60mg/L C₁₂H₂₅SO₄Na, pH=5). The temperature of the water bath was controlled at 85°C. To make the bath uniformly, the electric agitator was started at a speed of 300r/min. After 2 hours' plating time, the plated sample was cleaned by deionized water and then dried for testing.

Table 1. The formulation of the activation solution.

	Constituents	Quality (g/L)
Electric clean liquid	NaOH	25.0
	Na ₂ CO ₃	21.7
	Na ₃ PO ₄	50.0
	NaCl	2.4
No. 1 activation solution	HCl	25
	NaCl	140.1
No. 2 activation solution	Na ₃ C ₆ H ₅ O ₇ ·2H ₂ O	141.2
	H ₃ C ₆ H ₅ O ₇ ·H ₂ O	94.2
	NiCl ₂ ·6H ₂ O	3

2.2 Performance testing

The microstructure of Ni-P composite coating was observed by FEI scanning electron microscope. The magnification was 10000 times. Chemical composite coating samples were detected by using Panalytical X'Pert Powder type X-ray diffractometer (Incoming rays are $K\alpha$ rays, wavelength 1.54i; tube voltage 40kV; tube current 50mA; continuous scan scanning step length 0.02° , scanning range 10° to 90°). The surface hardness test of the coating was carried out on the MH-3 digital microscope hardness tester, with the load of $F=500\text{g}$, and the loading time was 10s. The adhesive force of each specimen was tested by WS-2005 adhesion automatic scratch tester. The dynamic load was 50 N, the scratch length was 2 mm, and the reciprocating time was 1. The wear resistance test was carried out on the SFT-1 material surface comprehensive tester. The grinding speed was 500 r / min, the running radius was 3 mm, the loading load was $F=320\text{g}$, and the wear time was 30 min.

3. Results and discussion

3.1 Chemical plating microstructure

Fig. 1 shows the effect of substrate roughness on the surface morphology of the coating. It can be seen from Fig. 1 that when the substrate roughness is $0.9635\mu\text{m}$, the spherical particles deposited on the surface of the coating are of different sizes, uneven distribution and obvious directivity, due to the severe surface roughness of the sample and the sharp height difference between peak and valley, and the scratches on the coating being not removed. In the process of electroless plating, the coating tends to deposit^[16-18] along the scratch direction, resulting in uneven surface morphology.

With the decrease of the roughness of matrix, the accumulation of spherical particles becomes more compact, distributed and uniform in size. When the roughness of the substrate is $0.0935\mu\text{m}$, the spherical particles are combined tight and the coverage rate is high, distributed in the shape of cauliflower, and the morphology was the best among the four samples. As the roughness decreases, the microstructure becomes denser and the scratches of the matrix are gradually covered.

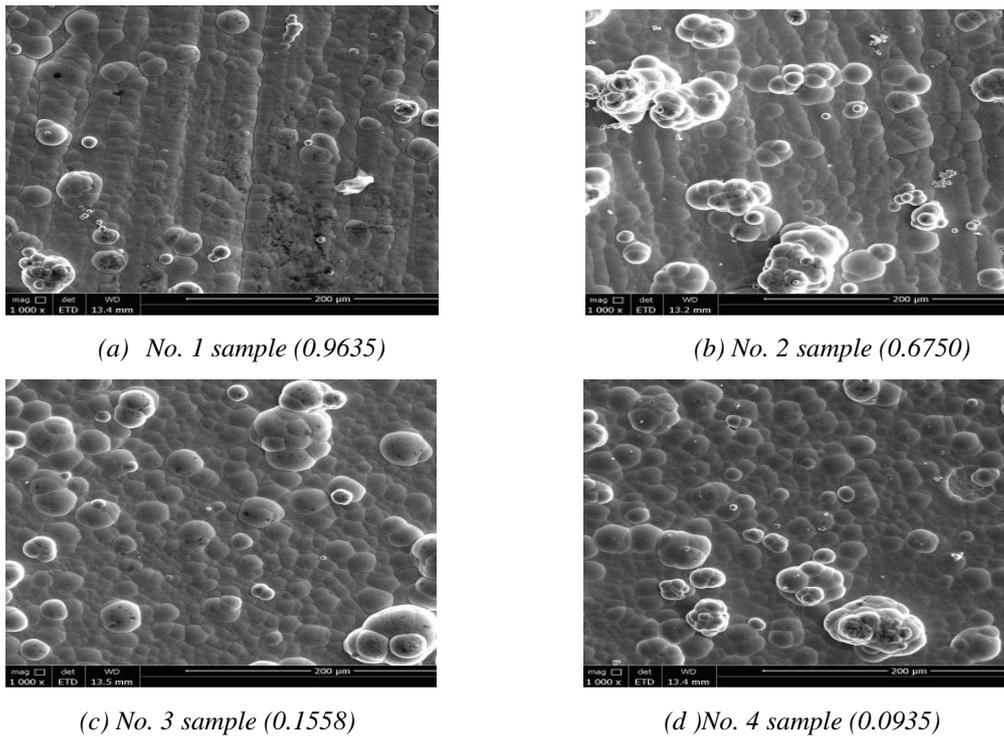


Fig. 1. Surface morphology of coatings obtained from different substrate roughness.

3.2 Phase analysis

Fig. 2 shows the XRD results of the Ni-P chemical composite coating. Sharp diffraction peaks appear in the range of 40° to 50° 、 50° to 55° and 75° to 80° when the roughness of matrix is 0.9635, 0.6750 and 0.1558, respectively. The peak appearing in the range of 40° to 50° is the diffraction peak of Ni(1 1 1); the peak appearing in the range of 50° to 55° is the diffraction peak of Fe(2 0 0); and the peak appearing in the range of 75° to 80° is the diffraction peak of Fe(2 1 1). From the analysis of 3.1, these four samples are founded to be coated with Ni-P on the surface, therefore, the appearance of the diffraction peaks of Fe on the XRD spectra is not due to the failure of the sample to be electroless plated successfully, it's due to the thin thickness of the coatings of the first three samples, which makes the diffraction peaks of Fe can be left on the XRD spectra. As is shown in the Fig. 2, with the decrease of roughness, the diffraction peaks of Fe tend to be less obvious. There are no diffraction peaks of Fe on the spectrum when the roughness of matrix is 0.0935, which indicates that the coating of sample No. 4 is thick and dense enough. The results above show that the smaller the roughness of the substrate is, the more uniform the microstructure of the coating is, therefore the Ni-P particles can be distributed on the substrate surface. The results above indicate that the smaller the substrate roughness, the thicker the coating and the better the coating's quality.

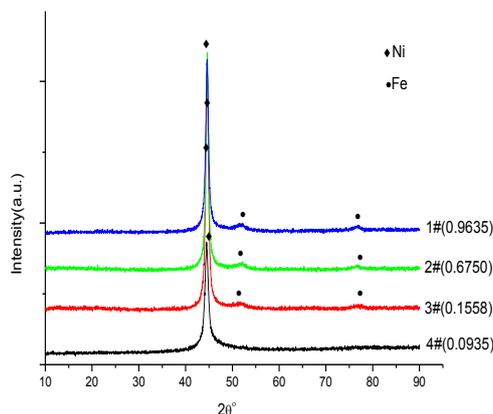


Fig. 2. X-ray diffraction spectra of coatings obtained from different substrate roughness.

3.3 Hardness of coating

As shown in Fig. 3, the hardness of coating decreases with the increase of substrate roughness. This is because the coating porosity is particularly sensitive to hardness, and uniform and dense microstructure leads to high hardness coating. This is consistent with the conclusion that with the increase of the roughness of the substrate, the surface quality of the coating becomes worse, and the microstructure becomes sparse.

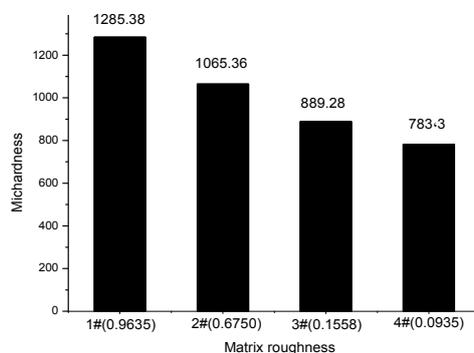


Fig. 3. Hardness of the coating with different matrix roughness.

3.4 Coat adhesion

Fig. 4 shows when the roughness is 0.9635 and the head load reaches 5.35N, the sound signal of cracking is emitted and the coating cracks and when the roughness is 0.0935, the coating will start to crack and the pressure reaches 32.9N. It shows that the lower the roughness is, the greater the critical load of cracking is, and the better the bonding between the coating and the substrate is. The increase in adhesion is due to the release of H atoms from the coating during the deposition process and the corresponding reduction of the internal stress of the coating, which makes the overall stress of the composite coating mainly reflect the compressive stress. Because the smaller the roughness of the coating is, the flatter the surface is, and the easier it is for diffusion to occur, which strengthens the coat adhesion. On the other hand, the adhesion is also related to the thickness of the coating. The smaller the roughness of the substrate, the thicker the

coating and the greater the adhesion, which is consistent with the result of the analysis in 3.2 that the smaller the roughness, the thicker the coating.

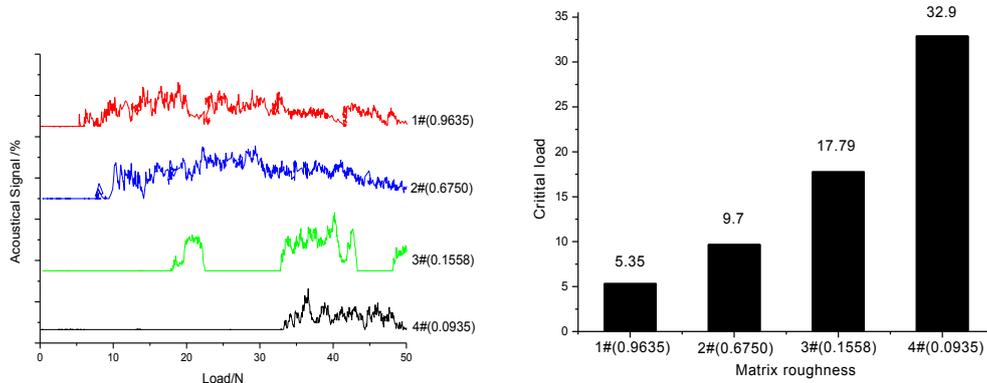


Fig. 4. The effect of different matrix roughness on the bonding strength of the coating.

3.5 Coating wear resistance

Fig. 5 shows the wear mass loss of coatings obtained from different matrix roughness and figure 6 if friction coefficient of coating obtained from different matrix roughness. From Fig. 5, it can be seen that with the increase of substrate roughness, the wear mass loss of coatings and the friction coefficient increases accordingly, which indicates the wear resistance of the coating becomes weakened. This is because the surface of the plating part with small matrix roughness is smooth, the interface is not obvious crack, the interface is relatively dense, the bonding force between the substrate and the coating is better^[19-20], it is not easy to be worn out, and the coating has good wear resistance^[21]. On the other hand, the phase structure of the coating is mainly amorphous Ni-P, and the amorphous alloy structure can achieve a solid self-lubrication effect. From the microscopic observation results, the smaller the roughness of the matrix, the more uniform distribution of Ni-P particles and the higher the coverage, the better the self lubrication effect of the deposited coating, therefore the better wear resistance, which is consistent with the result of the analysis in 3.2 that the smaller the roughness is, the thicker the coating is.

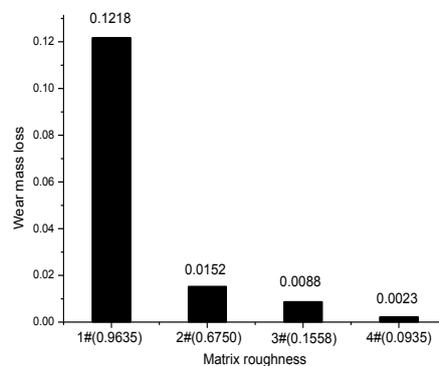


Fig. 5. Wear mass loss of coatings obtained from different matrix roughness.

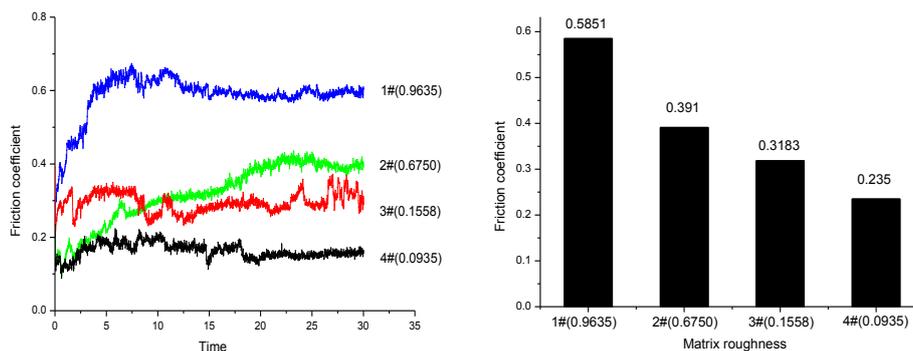


Fig. 6. Friction coefficient of coating obtained from different matrix roughness.

4. Conclusions

(1) When the roughness of the substrate is large, the spherical particles accumulated on the surface of the coating are of different sizes and have obvious directionality. The smaller the roughness of the matrix is, the more uniform the spherical particles are, the closer they are bonded, and the higher the coverage rate is, the denser the coating is.

(2) The phase structure of the coating is mainly amorphous Ni-P. The matrix Fe diffraction peak exists when the matrix roughness is too large, which indicates that the coating is thin, and with the decrease of the matrix roughness, the matrix Fe diffraction peak disappears and the Ni-P particles uniformly distribute on the substrate surface, hence the better coating quality.

(3) The adhesion between the coating and the substrate decreases with the increase of the roughness of the substrate. When the roughness is $0.0935 \mu\text{m}$, the adhesion between the coating and the substrate is the largest and the best.

(4) The hardness and wear resistance of the coating become worse with the increase of the substrate roughness. When the roughness is $0.0935 \mu\text{m}$, the hardness is the highest, the wear and friction coefficient is the smallest, and the wear resistance is the best.

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